C160-E009A

SHIMADZU
Excellence in Science

Thermal Analysis 60 Series

Application Data Book

Food and Pharmaceutical

Contents

1. Melting of Fats and Oils

1.1 Chocolate

■Explanation

DSC (Differential Scanning Calorimeters) analyzes melting processes of cooking oils. Cocoa butter, an ingredient of chocolate, exhibits 6 crystal structures, of which the type V-crystal is said to be stable against heat. Because the V-type crystal melts at around 34˚C, its existence in chocolate can be examined by DSC. Fig 1.1.1 shows the DSC curve of chocolate heated at 3˚C/min. Fig. 1.1.2 shows the DSC curve of the same chocolate when it is once melted, cooled to -50˚C for crystallization and then reheated. The peak at 30.4˚C has completely disappeared.

■Analytical Conditions

Instrument : DSC-60
Sample name : Chocolate
Sample weight : 22.87 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 3˚C/min

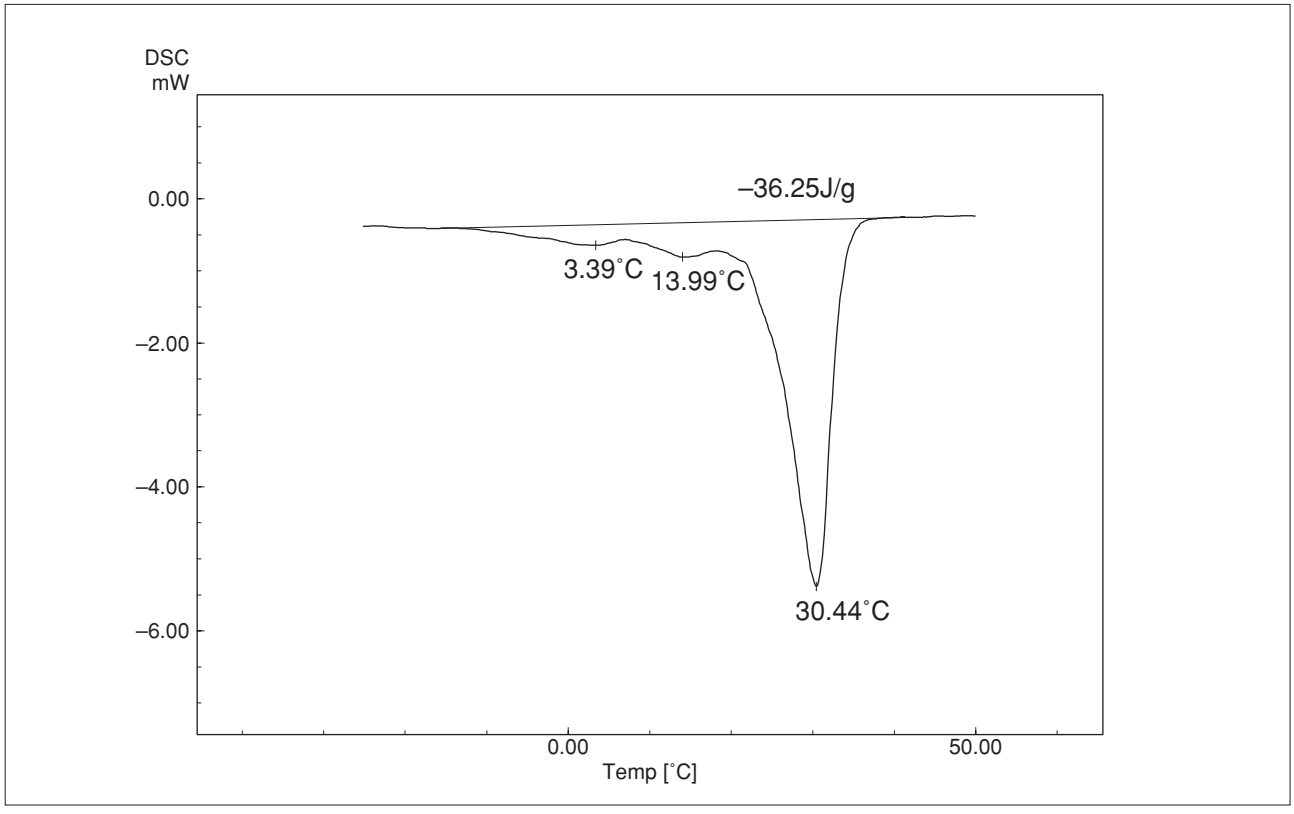

Fig. 1.1.1 First measurement of chocolate

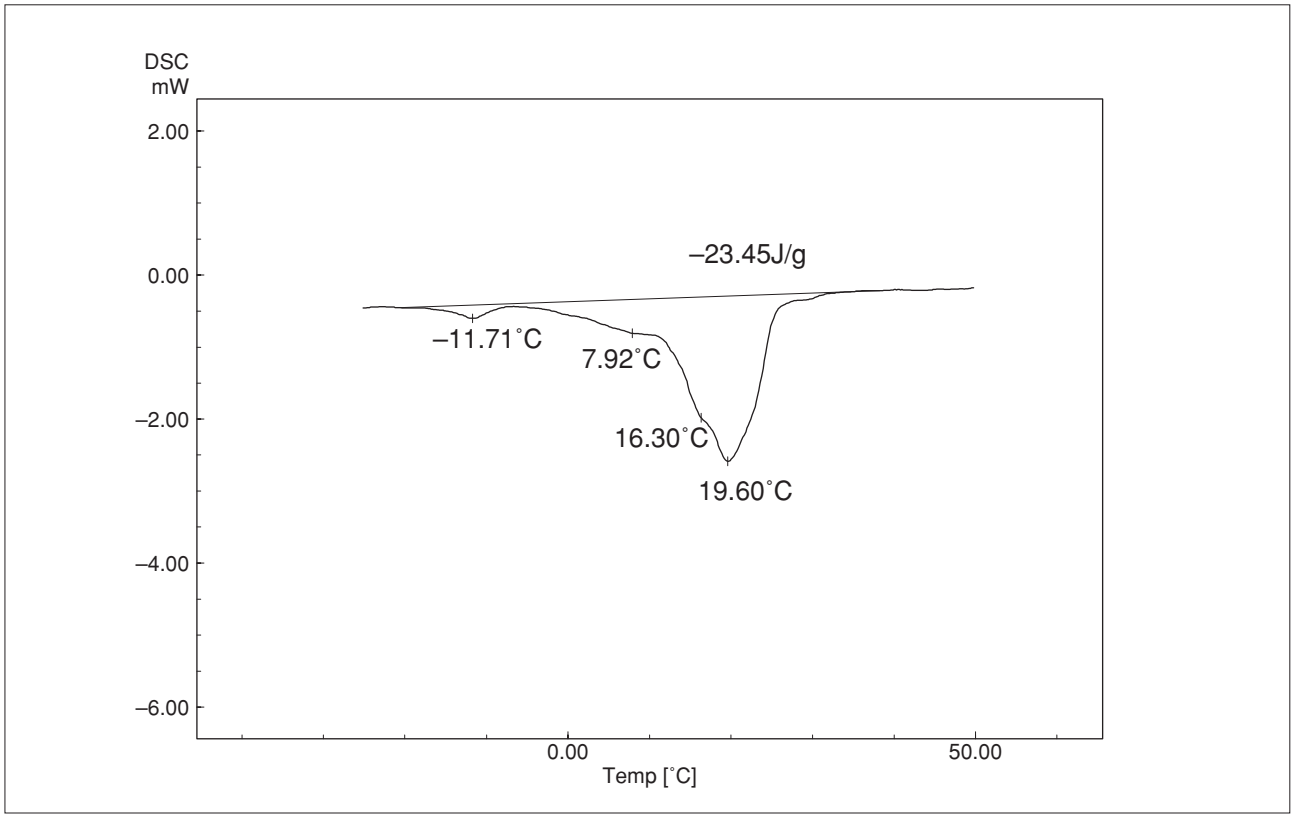

Fig. 1.1.2 Second measurement of chocolate

1.2 Olive Oil

■Explanation

This section shows the measurement of the melting of olive oil. The exothermic peak at -29.8˚C due to crystallization and the melting peak at -5.4˚C are clearly observed.

■Analytical Conditions

Instrument : DSC-60
Sample name : Olive oil
Sample weight : 18.2 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 3˚C/min

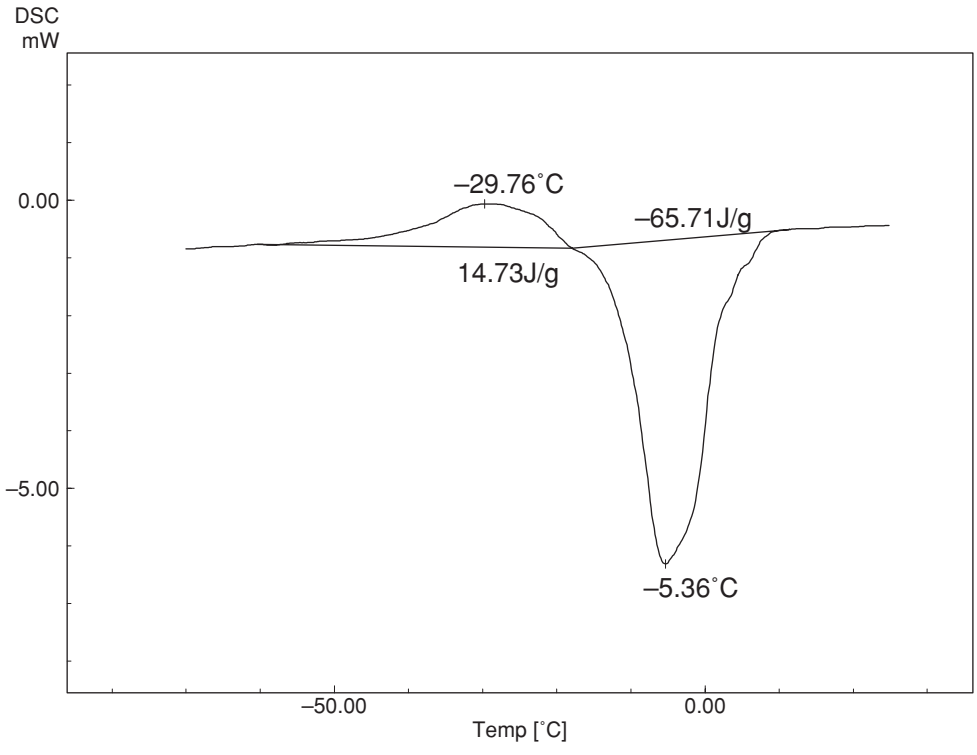

Fig. 1.2.1 DSC curve for olive oil

1.3 Margarine

■Explanation

The fats and oils in margarine include triglycerides consisting of various types of fatty acids. With the margarine analyzed, melting peaks of various triglycerides are visible across a wide range of temperatures (from -33 to +22.4˚C). In this example, the partial area analysis program was used to calculate the heat of fusion from peak-start to specified temperatures.

■Analytical Conditions

Instrument : DSC-60
Software used : Partial Area Analysis Program
Sample name : Margarine
Sample weight : 19.57 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 3˚C/min

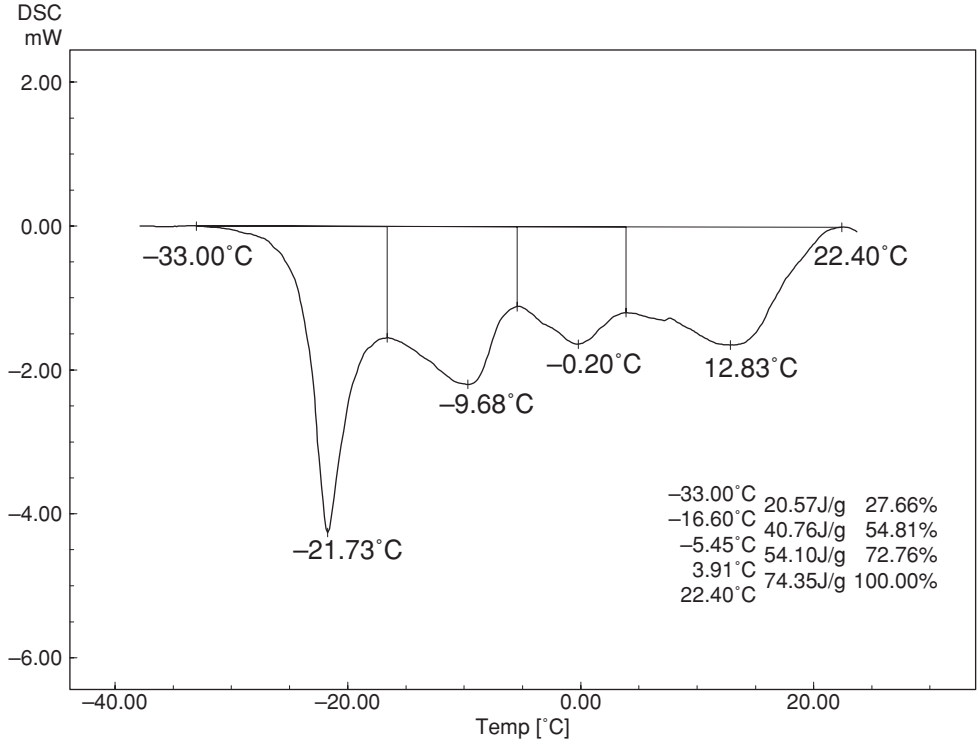

Fig. 1.3.1 DSC curve for margarine

2. Degradation of Cooking Oil by Oxidation

2.1 Salad Oil

■Explanation

Properties of cooking oil, such as taste, smell and color, change with long term storage or heating. The main cause of these changes is oxidation. TG (Thermogravimetry) can measure the changes in mass due to oxidation. The results of TG can be used to determine storage conditions, estimate shelf life and evaluate antioxidants. In this example, salad oil was heated in oxygen. Absorption of oxygen began at 153.5˚C and the weight increased by 0.95%. The surface area of the sample influences the weight increase. This test used a special cell of 10 ø diameter.

■Analytical Conditions

Instrument : TGA-50
Sample name : Salad oil
Sample weight : 22.85 mg
Atmospheric gas : Oxygen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 2˚C/min

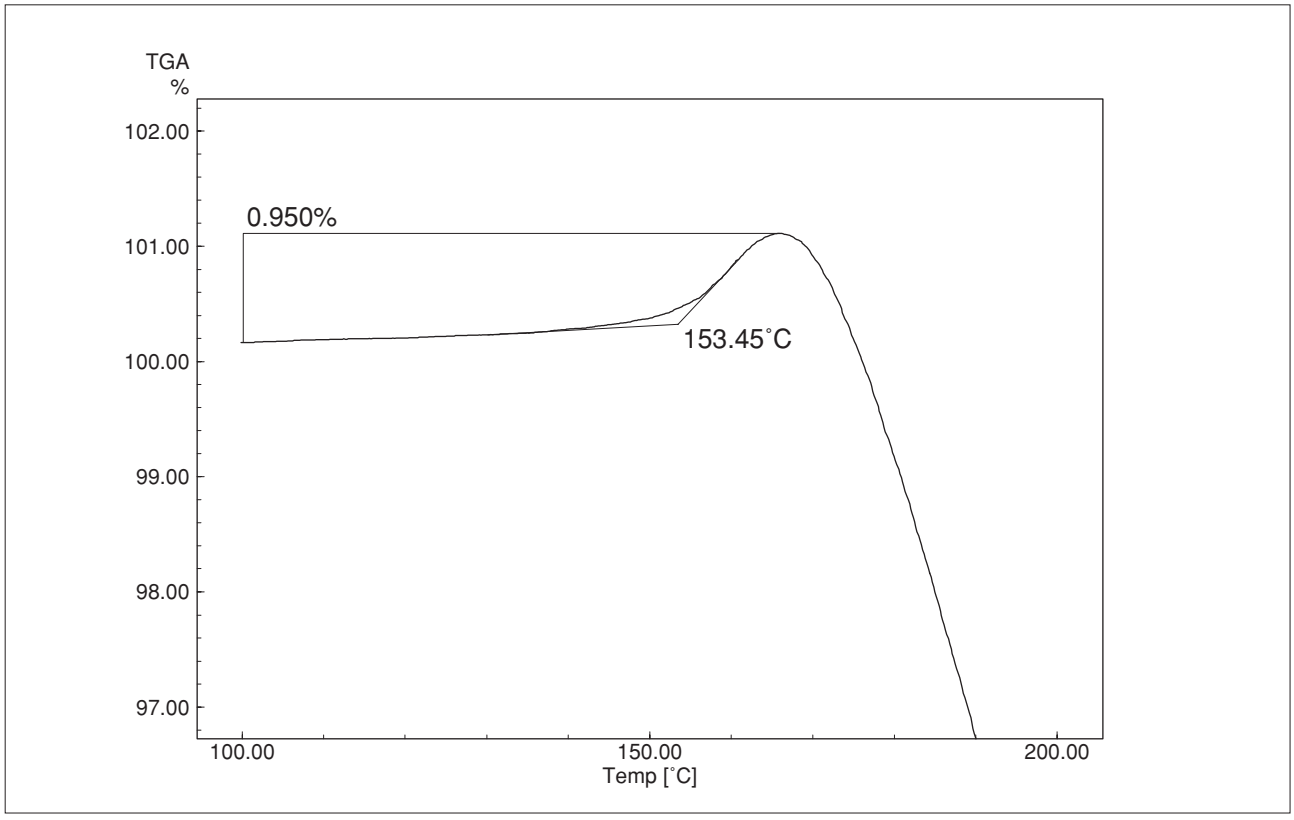

Fig. 2.1.1 TG curve for salad oil in oxygen

2.2 Sesame Oil

■Explanation

In this example, sesame oil was heated in oxygen. Absorption of oxygen began at 152.4˚C and the weight increased by 0.31%.

■Analytical Conditions

Instrument : TGA-50
Sample name : Sesame oil
Sample weight : 23.532 mg
Atmospheric gas : Oxygen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 2˚C/min

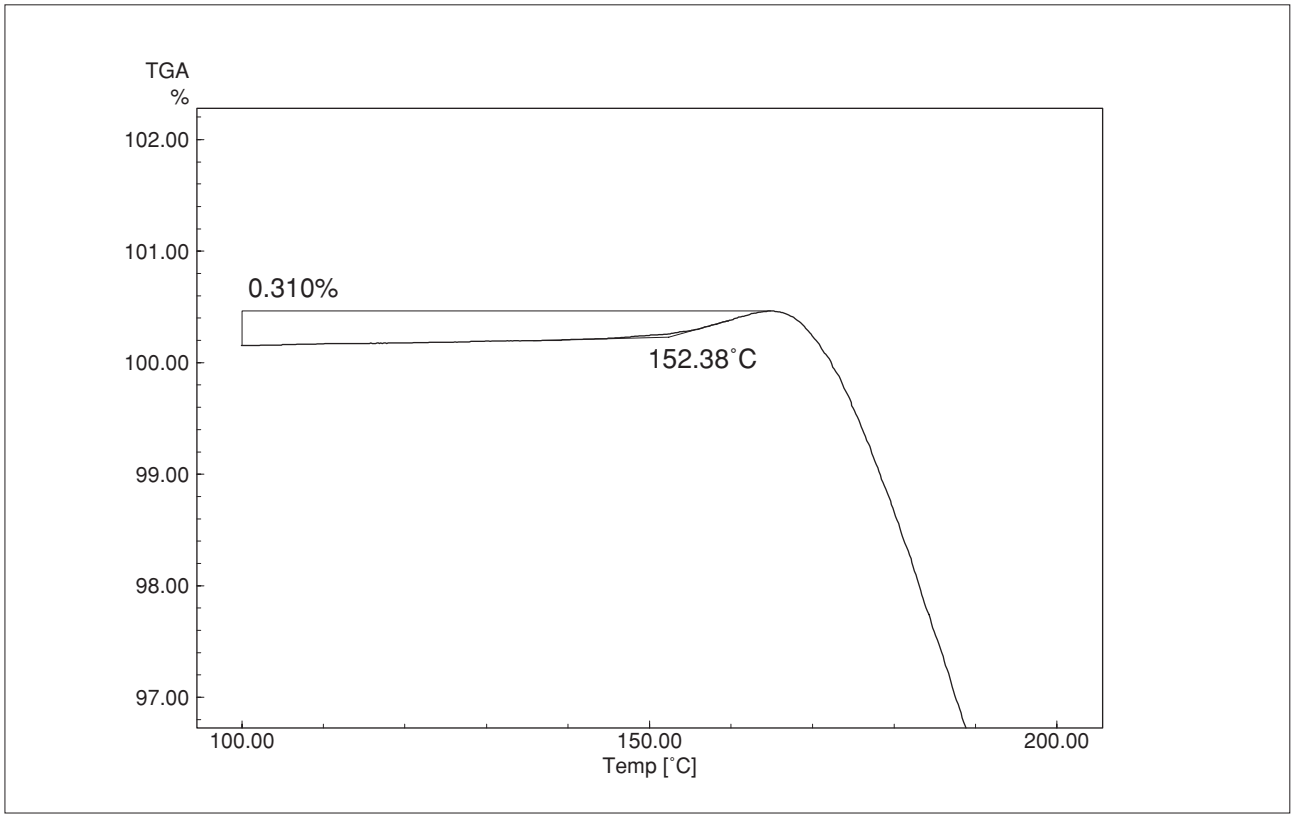

Fig. 2.2.1 TG curve for sesame oil in oxygen

3. Gelatinization of Starch

3.1 Flour

■Explanation

Starches gelatinize when heated with water. The gelatinization reaction can be analyzed by DSC because it is accompanied by endothermic reaction. This test measured flour starch (17.4%).

■Analytical Conditions

Instrument : DSC-60
Sample name : Flour
Sample weight : 4.21 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 5˚C/min

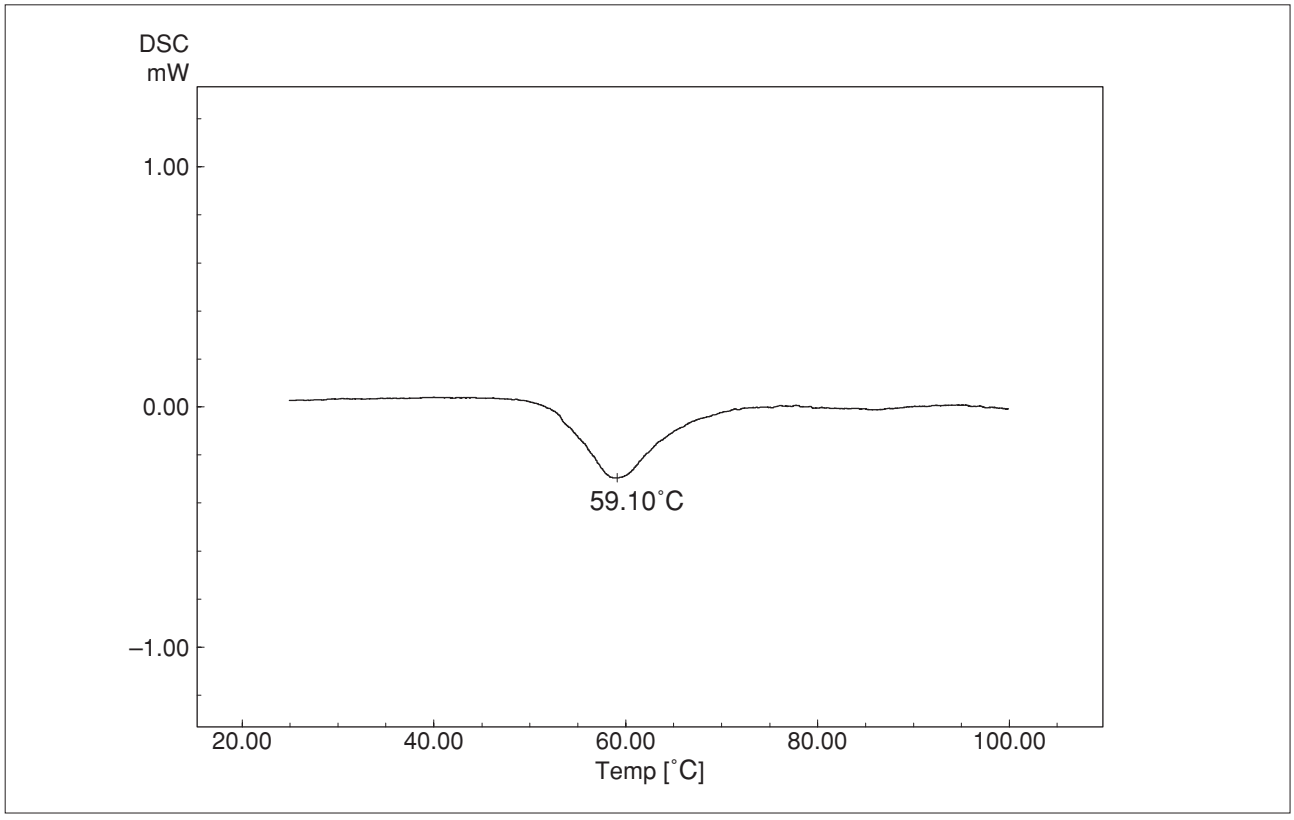

Fig. 3.1.1 Gelatinization temperature of wheat flour (17.4%)

3.2 Corn

■Explanation

This test measured cornstarch (19.9%). The gelatinization temperature of starch varies with additions such as sucrose or salt.

■Analytical Conditions

Instrument : DSC-60
Sample name : Corn
Sample weight : 4.97 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 5˚C/min

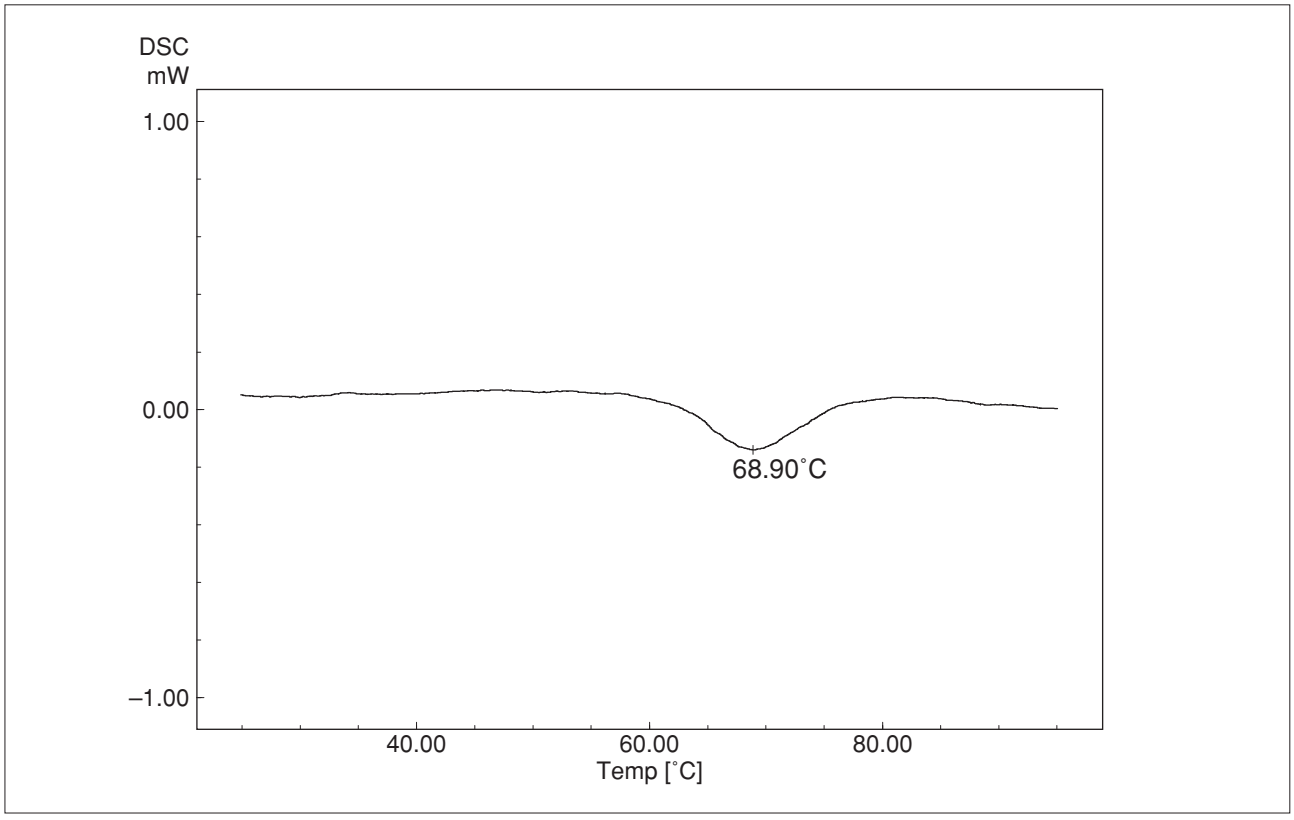

Fig. 3.2.1 Gelatinization temperature of corn (19.9%)

4. Retrogradation of Starch

4.1 Bread

■Explanation

When starch is heated with water for gelatinization and then let stand, the association and rearrangement of amylopectin in swelled starch particles proceed to gradually harden the particles. This process is called the retrogradation of starch. This example shows the measurement of bread. Fig. 4.1.1 shows freshly baked bread, Fig. 4.1.2 bread one day after baking, and Fig. 4.1.3 bread three days after baking. The peak area increases with the increase in the storage period.

■Analytical Conditions

Instrument	: DSC-60
Sample name	: Bread
Sample weight (Fig. 4.1.1)	: 13.25 mg
(Fig. 4.1.2)	13.85 mg
(Fig. 4.1.3)	12.28 mg
Atmospheric gas	: Nitrogen
Gas flow rate	: 30 mL/min
[Temperature program]	
Heating rate	: 10°C/min

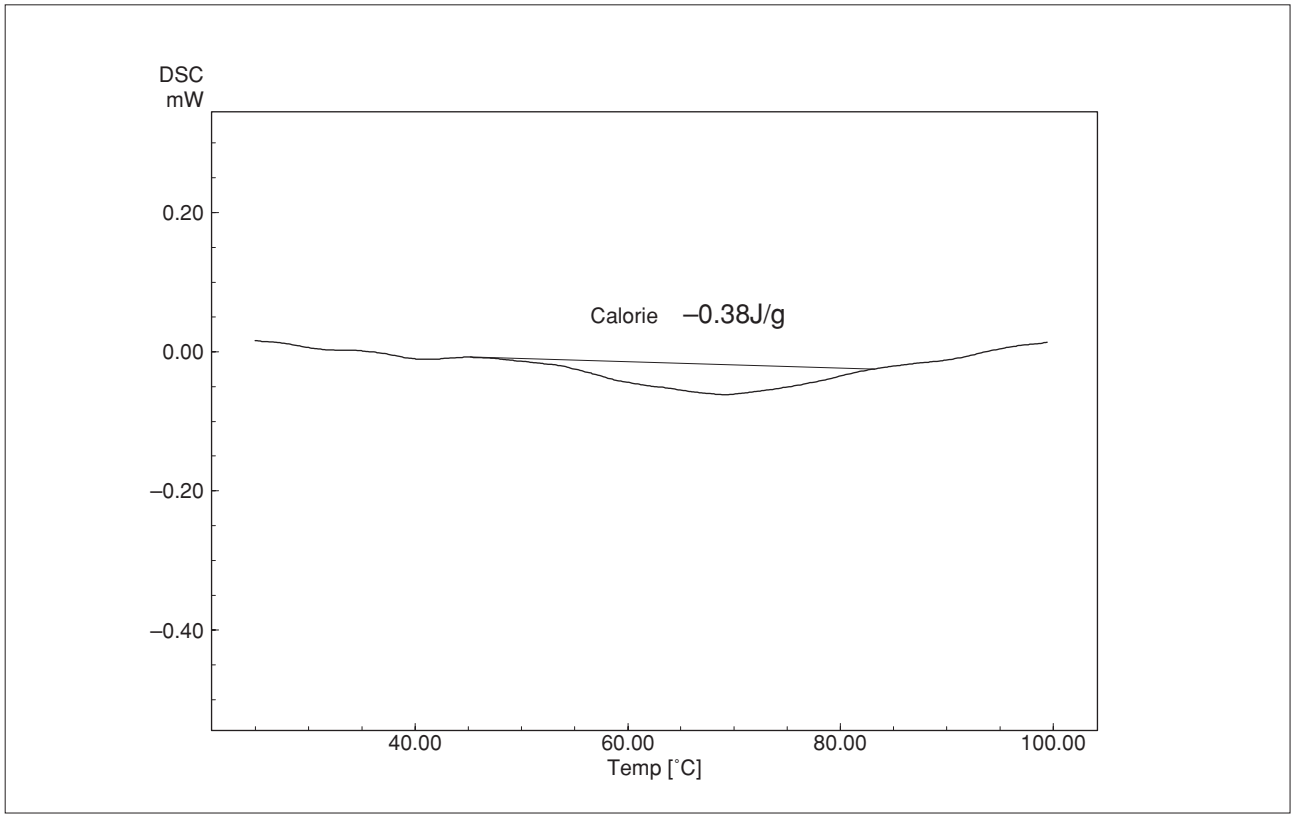

Fig. 4.1.1 DSC curve for freshly baked bread

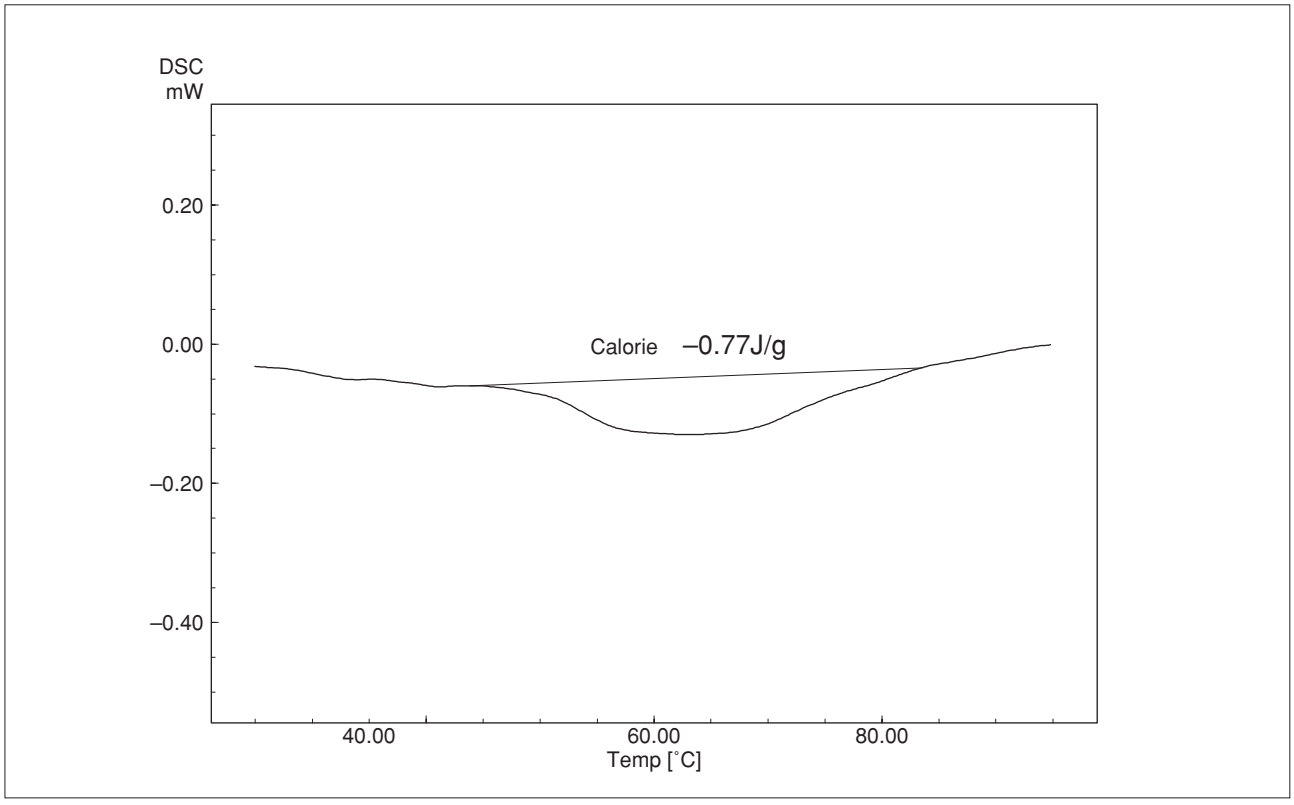

Fig. 4.1.2 DSC curve for bread one day after baking

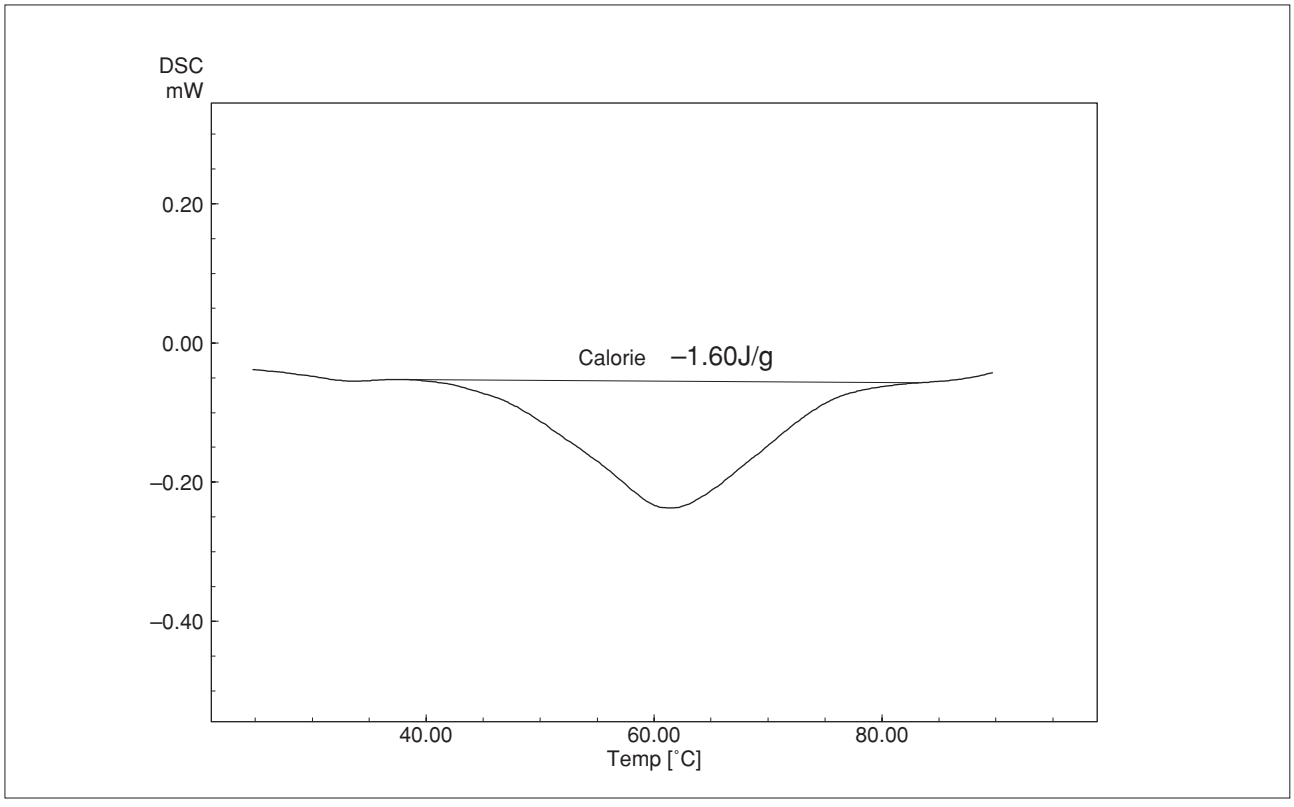

Fig. 4.1.3 DSC curve for bread three days after baking

5. Measurement of Moisture in Starch Gel

5.1 Sweet Potato Starch Gel

■Explanation

In starch gel, weakly bonded free water and strongly bonded bound water exist. The melting point of the water varies depending on the bonding conditions. The peak at -8.7˚C is the melting of the bound water and -3.8˚C the free water.

■Analytical Conditions

Instrument : DSC-60
Sample name : Sweet Potato Starch Gel
Sample weight : 11.98 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 2˚C/min

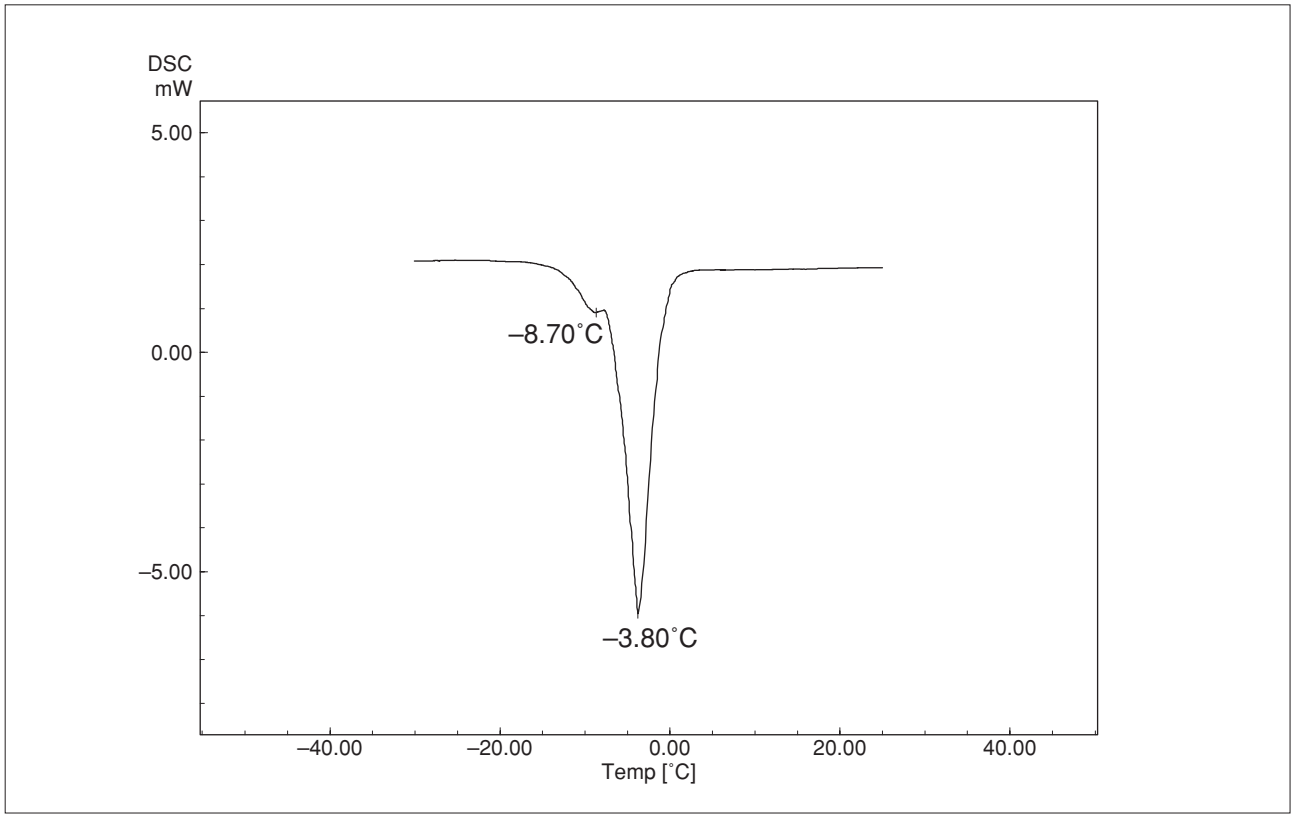

Fig. 5.1.1 Melting of water in sweet potato starch gel

5.2 Flour Starch Gel

■Explanation

Wheat flour tests showed bound water melting at -9.8˚C and free water at -4.3˚C.

■Analytical Conditions

Instrument : DSC-60
Sample name : Flour Starch Gel
Sample weight : 12.12 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 2˚C/min

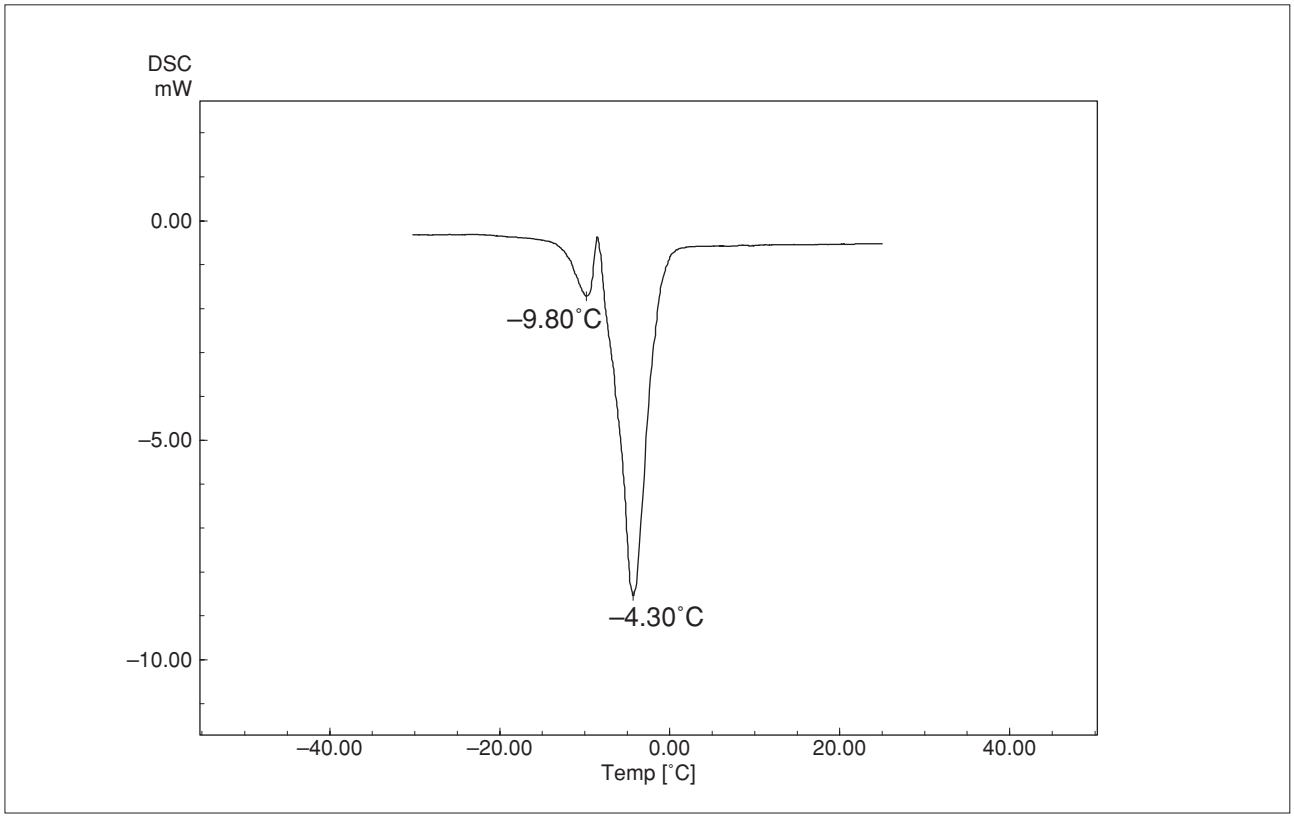

Fig. 5.2.1 Melting of water in flour starch gel

6. Liquor

6.1 Whiskey

■Explanation

Whiskey was sealed in a hermetic cell and measured by DSC. The exothermic peak at -74.2˚C is due to crystallization. The peak at -65.9˚C shows the melting of ethanol, -49.8˚C the eutectic point of ethanol and water, and -25.6˚C the melting of water. The height of the eutectic peak varies depending on the storage period of the malt.

■Analytical Conditions

Instrument : DSC-60
Sample name : Whiskey
Sample weight : 13.7 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 10˚C/min

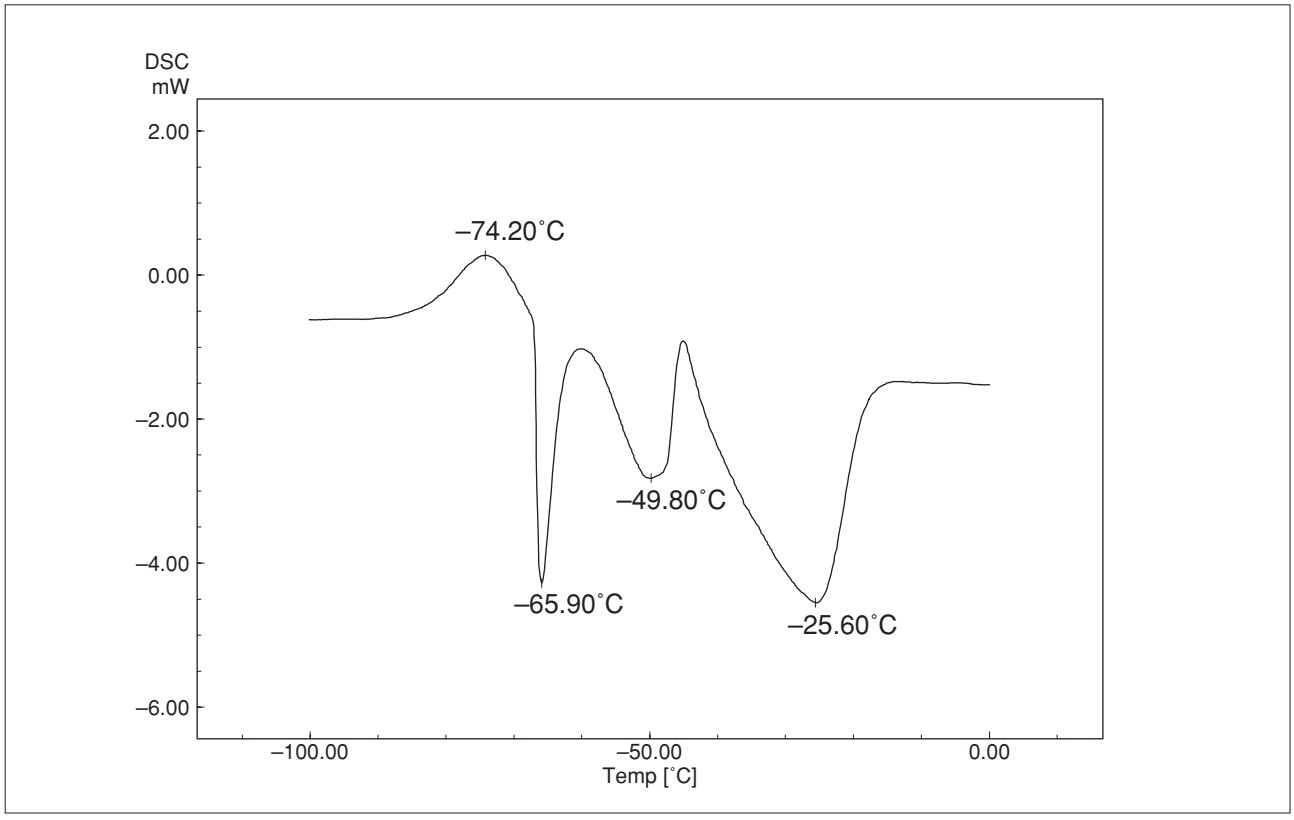

Fig. 6.1.1 DSC curve for whiskey

6.2 Brandy

■Explanation

Brandy was sealed in a hermetic cell and measured by DSC.

■Analytical Conditions

Instrument : DSC-60
Sample name : Brandy
Sample weight : 9.11 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 10°C/min

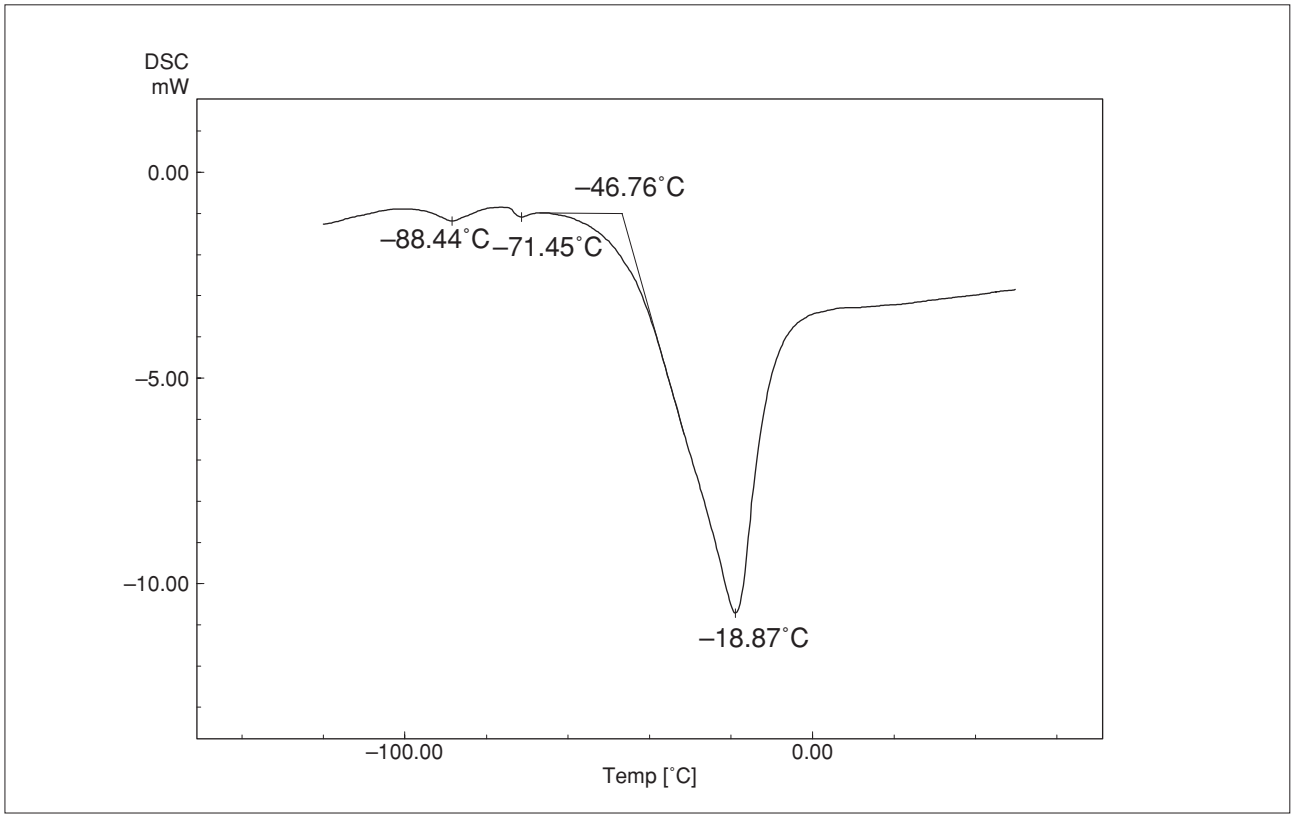

Fig. 6.2.1 DSC curve for brandy

7. Fish Meat

7.1 Fresh Carp Meat

■Explanation

DSC analyzed the freshness of carp meat. With the most fresh carp meat, an exothermic peak at 42.8˚C and endothermic peaks at 56.3˚C and 75.8˚C were observed (Fig. 7.1.1). With carp meat after 6 hours, a minute exothermic peak at 37.0˚C and endothermic peaks at 54.3˚C and 75.7˚C were observed (Fig. 7.1.2). With carp meat after 24 hours, endothermic peaks at 42.3˚C, 55.1˚C and 73.1˚C were observed but no exothermic peak was observed (Fig. 7.1.3). The endothermic peaks at 42˚C and around 73˚C are due to the denaturation of myosin and actin, respectively. The exothermic peak at around 40˚C corresponds to the shrinkage of myosin and actin caused by ATP remaining in the fish meat. The amount of ATP remaining varies with the storage period.

■Analytical Conditions

Instrument	: DSC-60
Sample name	: Carp Meat
Sample weight (Fig. 7.1.1)	: 25.4 mg
(Fig. 7.1.2)	24.5 mg
(Fig. 7.1.3)	26.5 mg
Atmospheric gas	: Nitrogen
Gas flow rate	: 30 mL/min
[Temperature program]	
Heating rate	: 5˚C/min

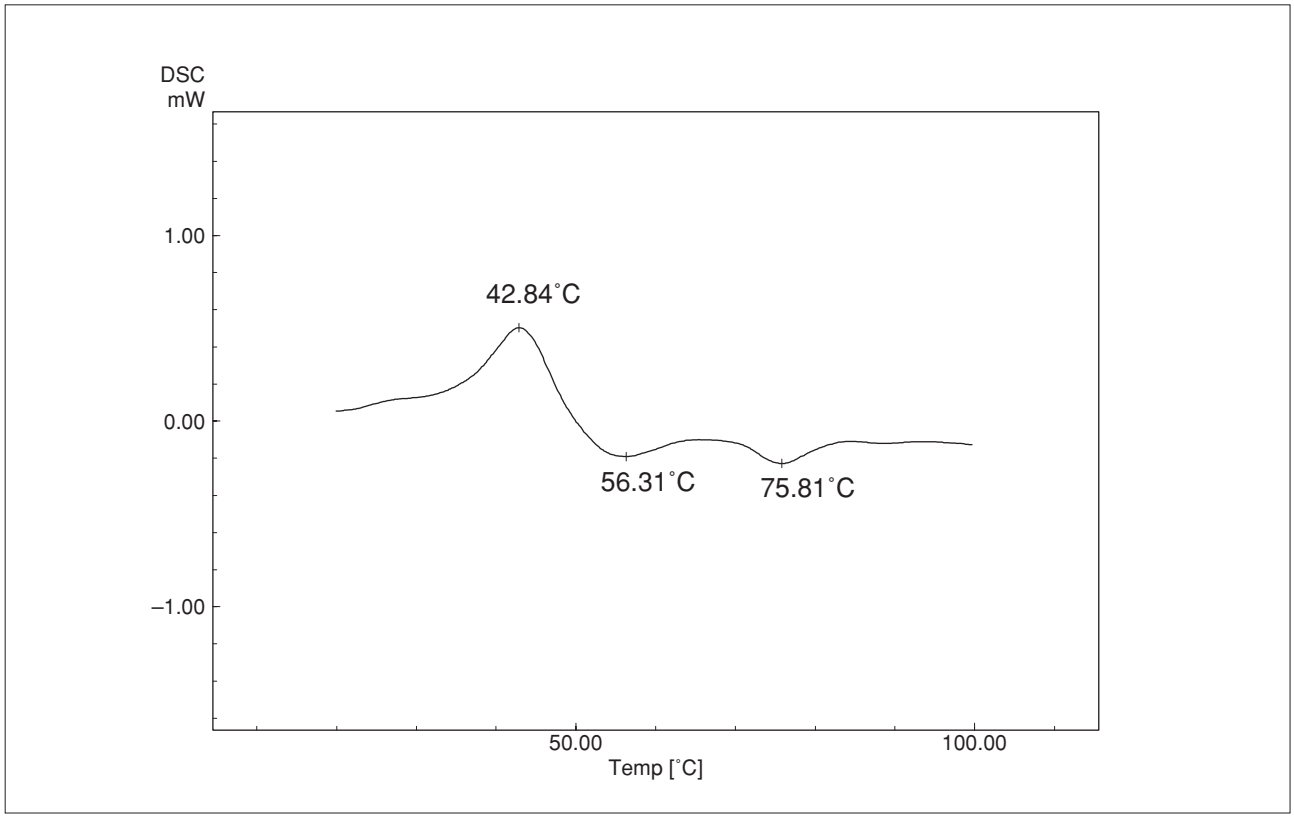

Fig. 7.1.1 DSC curve for fresh fish meat

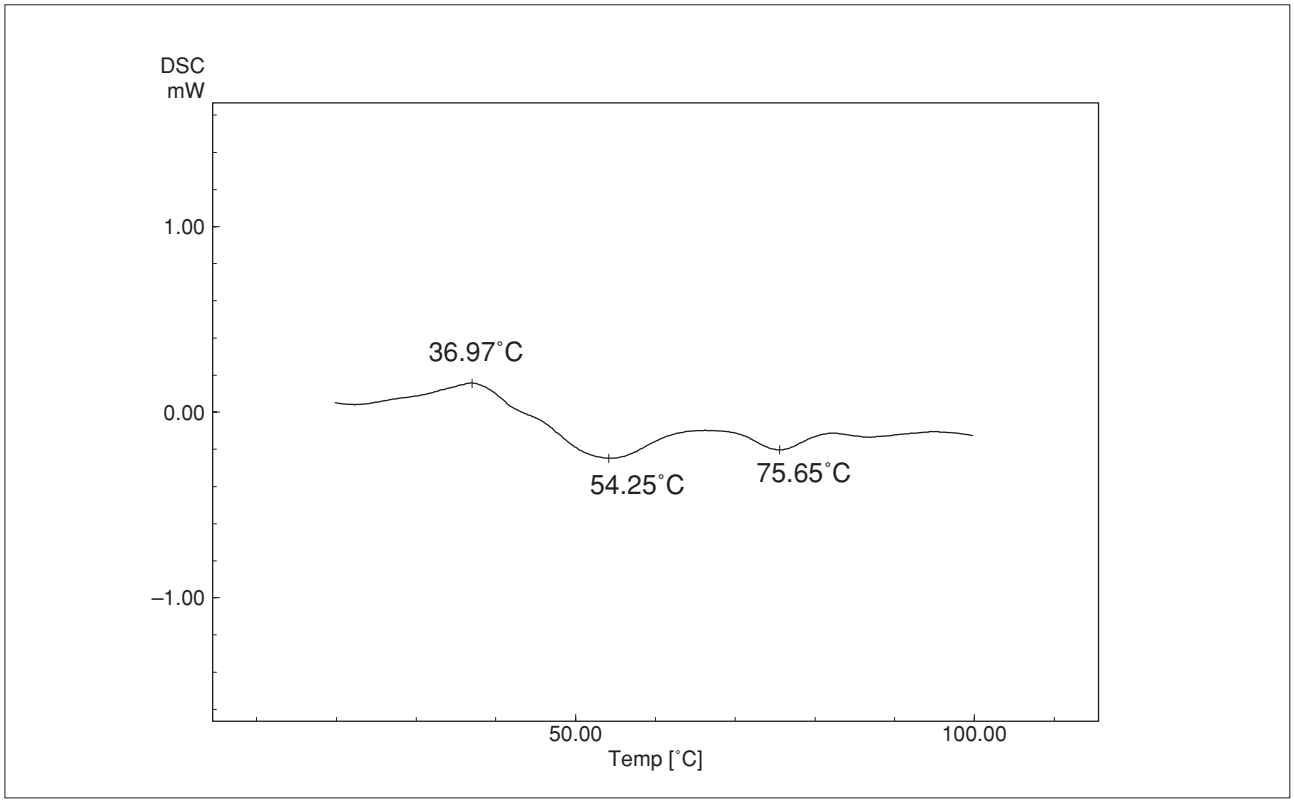

Fig. 7.1.2 DSC curve for fish meat after 6 hours

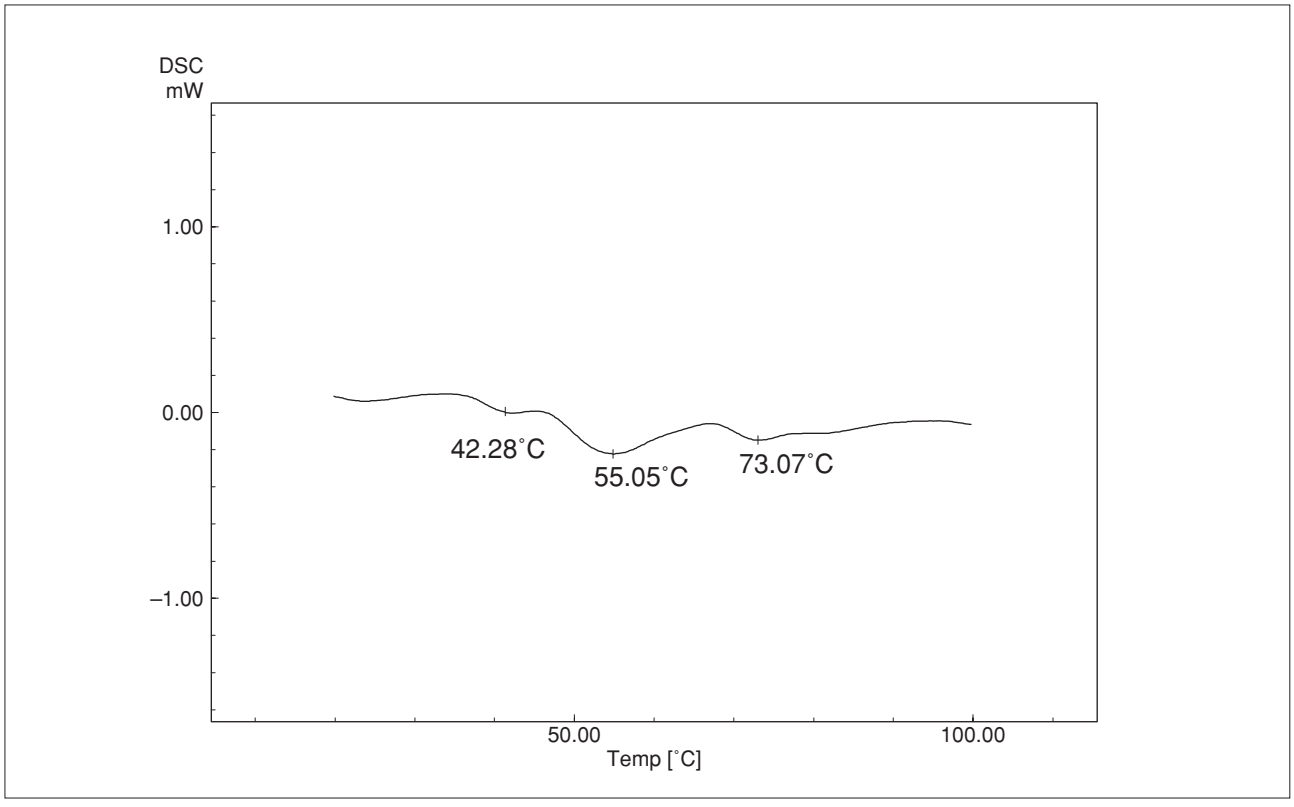

Fig. 7.1.3 DSC curve for fish meat after 24 hours

8. Water of Crystallization and Free Water

8.1 Measurement of Saccharin Sodium

■Explanation

When sweetener saccharine sodium is analyzed by TG two-stage weight loss due to dehydration is observed (Fig. 8.1.1). Normally, water removed below 100˚C is free water and water removed at 100˚C or more is water of crystallization. In actuality, however, some water of crystallization is removed below 100˚C. DSC measurement strictly distinguishes between free and water of crystallization. When the sample is sealed in a pressure-tight hermetic cell and measured by DSC, water exhibiting an endothermic peak is water of crystallization and water without the endothermic peak is free water. Two endothermic peaks were observed with saccharine sodium (Fig. 8.1.2). The peak at 61.6˚C corresponds to water of crystallization.

■Analytical Conditions

Instrument (Fig. 8.1.1) : TGA-50
(Fig. 8.1.2) DSC-60
Sample name : Carp Meat
Sample weight (Fig. 8.1.1) : 17.2 mg
(Fig. 8.1.2) 14.1 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 10˚C/min
(For both TGA and DSC)

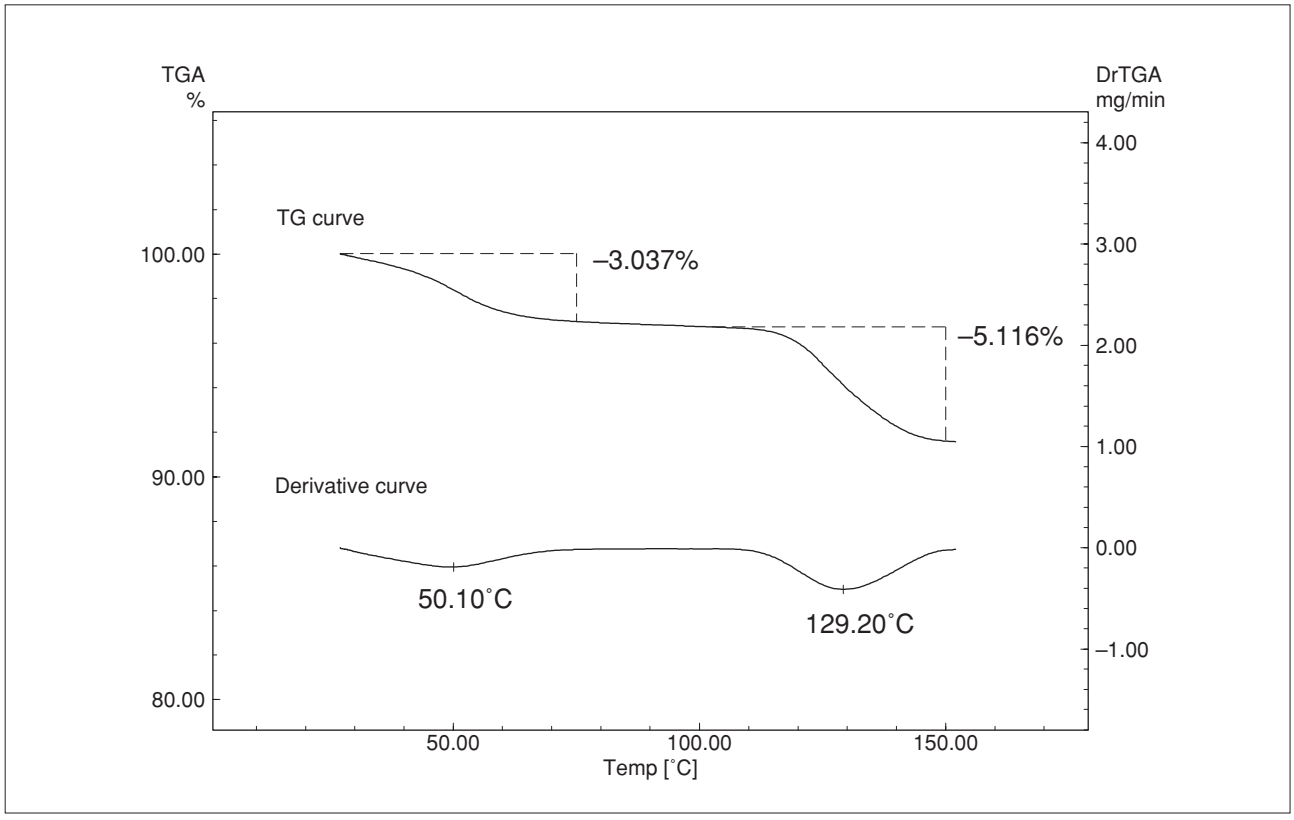

Fig. 8.1.1 TG-DTG curve for saccharine sodium

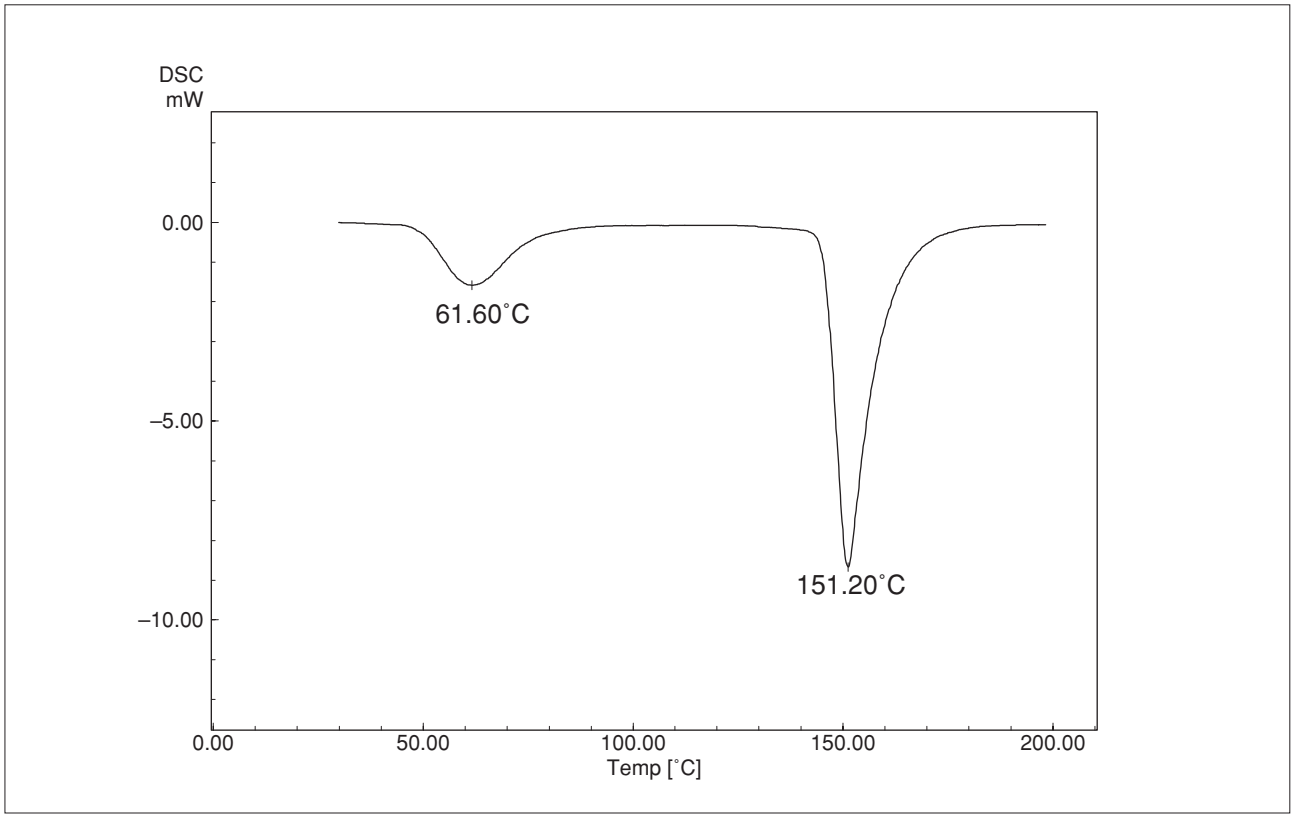

Fig. 8.1.2 DSC curve for saccharine sodium

9. Investigation of Freeze Drying Conditions

9.1 30% Glucose Aqueous Solution

■Explanation

DSC can analyze the relationship between the concentration and freezing conditions of a glucose aqueous solution.

In this example, the glucose has partially transformed to amorphous state. The minute peak at -62.7°C shows glass transition, and the exothermic peak at -47.3°C crystallization. The large endothermic peak at -17.6°C corresponds to melting.

■Analytical Conditions

Instrument : DSC-60
Sample name : 30% glucose aqueous solution
Sample weight : 8.0 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 10°C/min

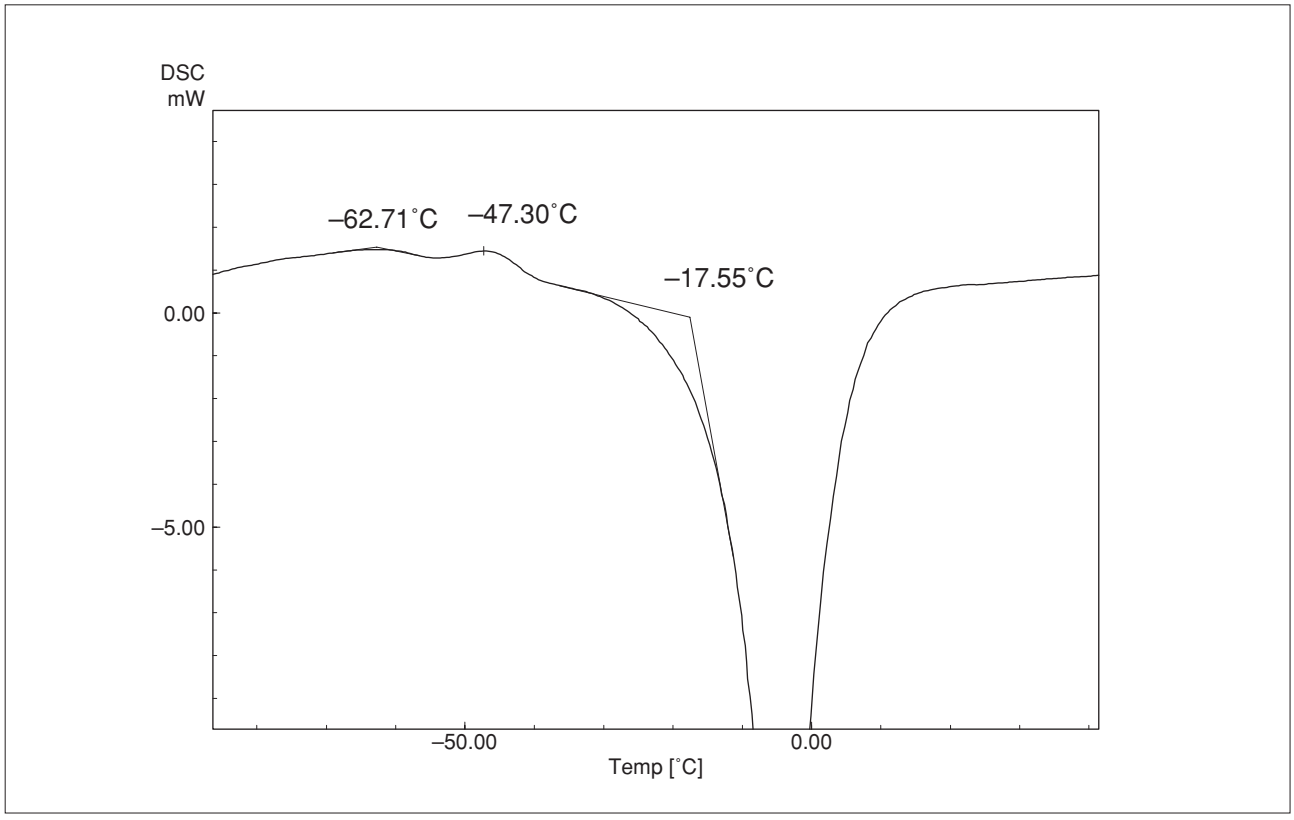

Fig. 9.1.1 DSC curve for 30% glucose aqueous solution

9.2 60% Glucose Aqueous Solution

■Explanation

The change at -89.2˚C shows glass transition and the exothermic peak at -36.8˚C crystallization. Because the areas of the crystallization peak at -36.8˚C and the melting peak at -22.7˚C are approximately equal, it is conceivable that the sample has mostly transformed to the amorphous state after freezing.

■Analytical Conditions

Instrument : DSC-60
Sample name : 60% glucose aqueous solution
Sample weight : 8.0 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 10˚C/min

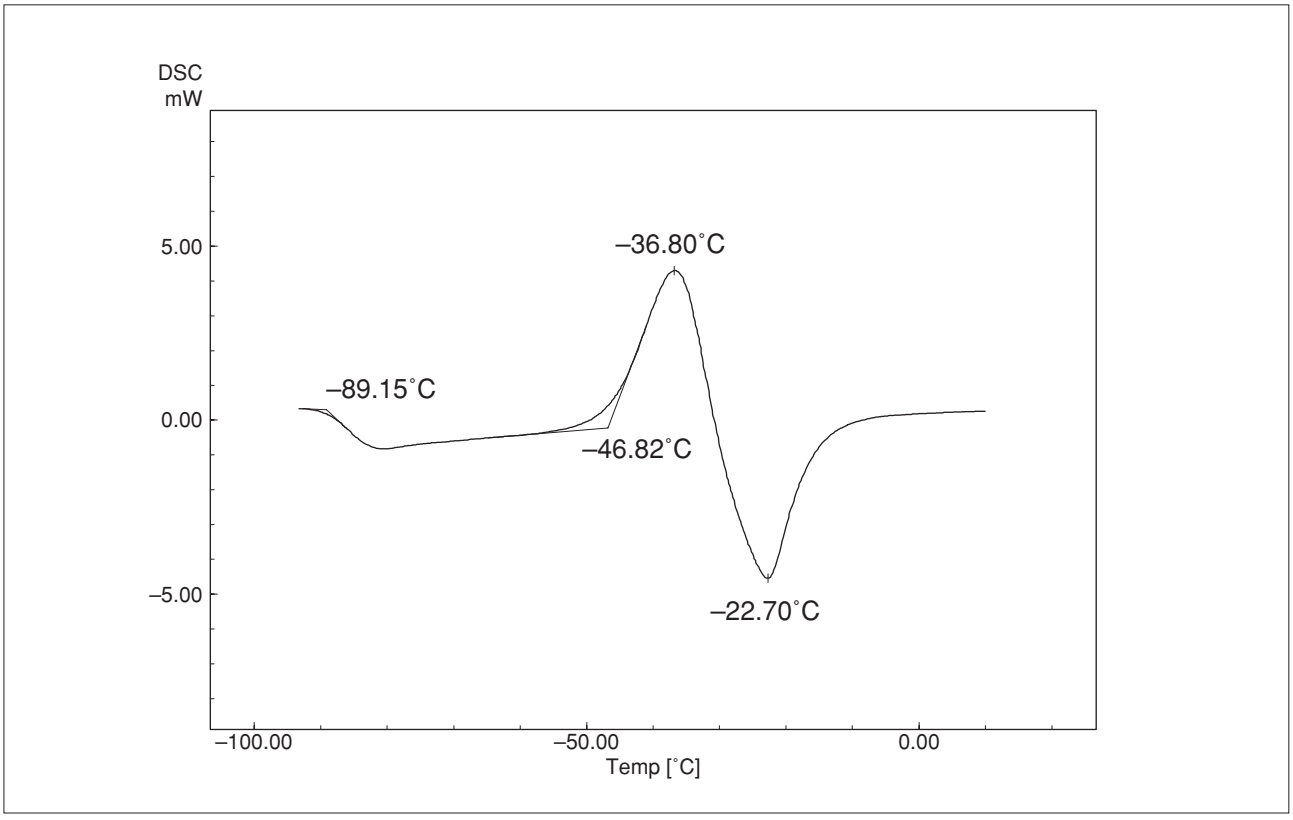

Fig. 9.2.1 DSC curve for 60% glucose aqueous solution

9.3 3% Salt Solution

■Explanation

With a 3% salt solution, a eutectic peak was detected at -23.6˚C. Generally, freeze drying is properly done at or below the eutectic temperature.

■Analytical Conditions

Instrument : DSC-60
Sample name : 3% salt solution
Sample weight : 6.99 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 10˚C/min

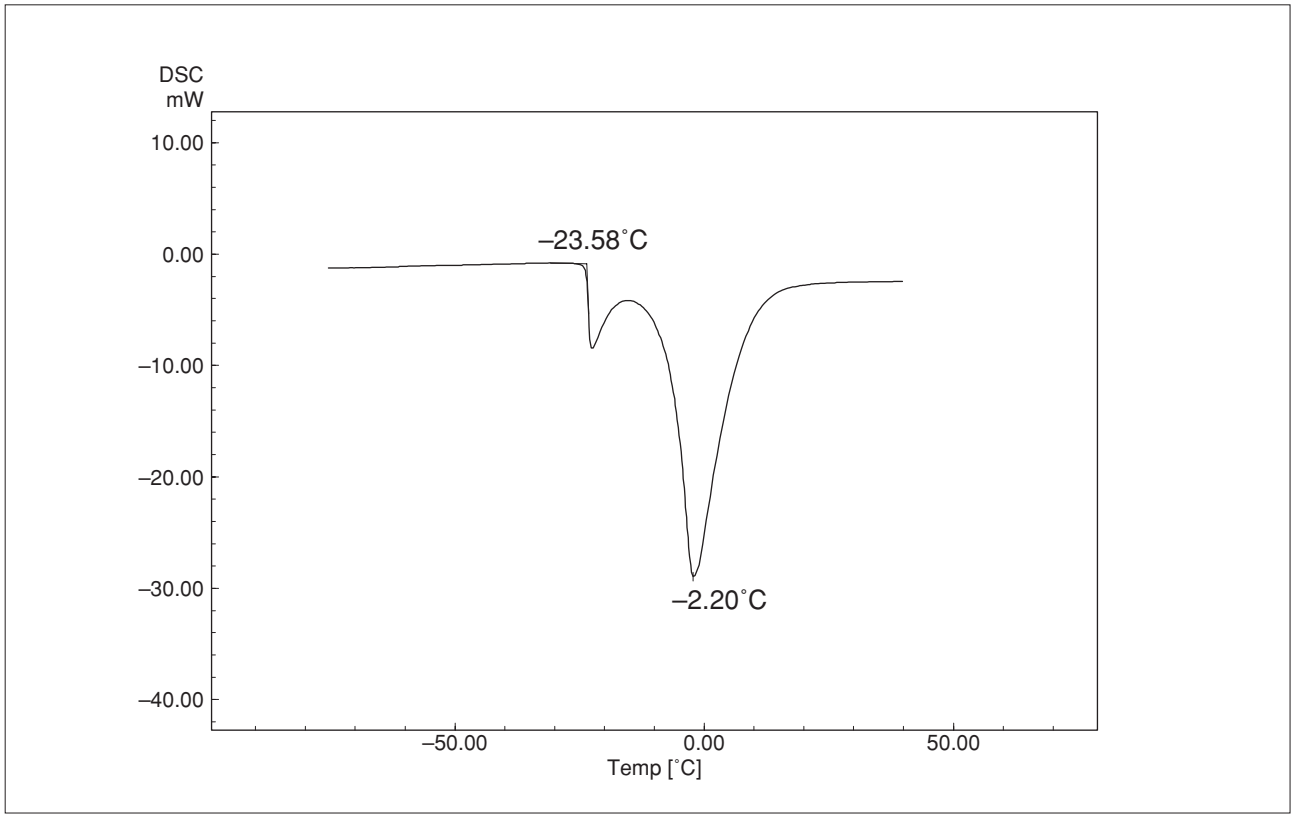

Fig. 9.3.1 DSC curve for 3% salt solution

10. Melting of Pharmaceuticals

10.1 Aspirin

■Explanation

Melting point and melting heat are fundamental thermophysical characteristics of pharmaceuticals and DSC can determine them. This example shows measurement of acetylsalicylic acid (aspirin), which has antipyretic analgesic and anti-inflammatory effects.

■Analytical Conditions

Instrument : DSC-60
Sample name : Aspirin
Sample weight : 3.09 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 5˚C/min

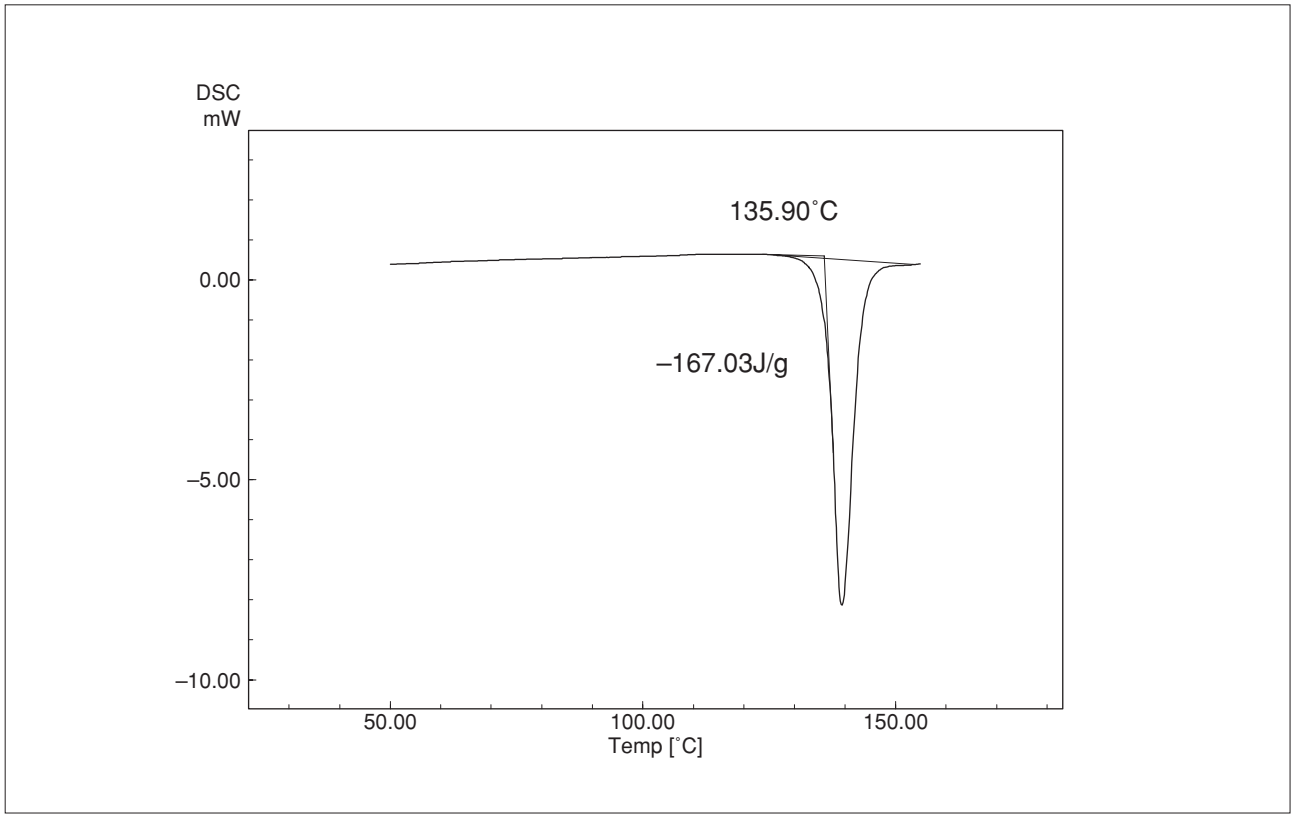

Fig. 10.1.1 DSC curve for aspirin

10.2 Polyethylene Glycol

■Explanation

This example shows the measurement of the melting point of polyethylene glycol (PEG) with various molecular weights. As the molecular weight increases, the melting point shifts to a higher temperature.
(The number added to the sample name indicates the molecular weight.)

■Analytical Conditions

Instrument : DSC-60 and TAC-60i
Sample name : PEG
Atmospheric gas : Nitrogen
Gas flow rate : 50 mL/min
[Temperature program]
Heating rate : 10°C/min
Hold temperature : 100°C

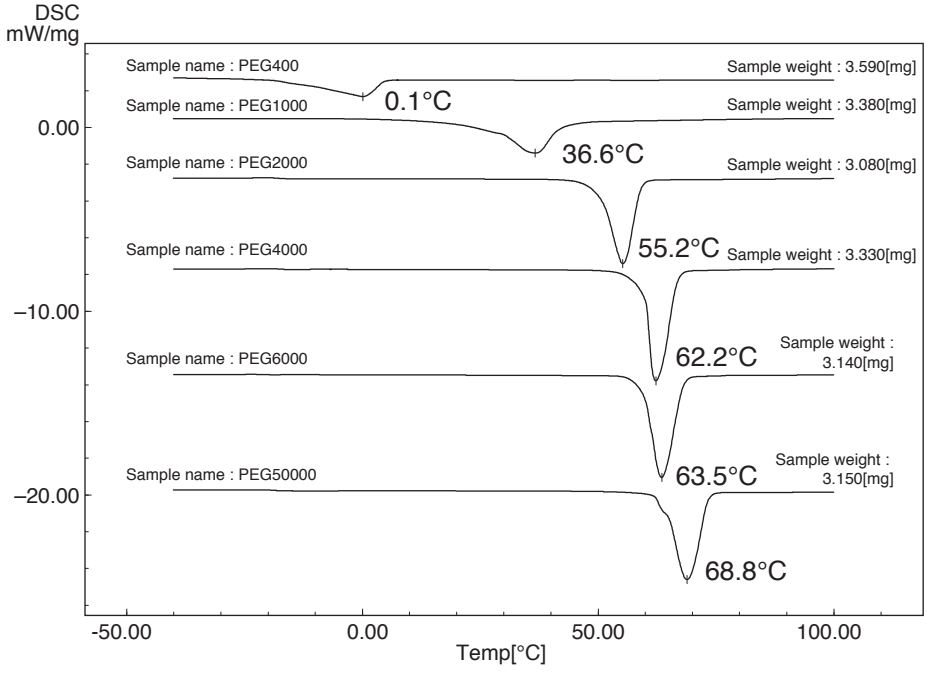

Fig. 10.2.1 DSC curve for polyethylene glycol

11. Investigation of Crystal Polymorphism –

11.1 Sulfathiazole

■Explanation

Generally, solubility of a substance differs depending on its crystal form. Therefore, crystal polymorphism is an important issue in pharmaceutical fields. Polymorphism is easily measured with DSC. Fig. 11.1.1 shows endothermic peaks at 168.3°C and 202.2°C. However, in Fig. 11.1.2, where sulfathiazole was heated to 185°C, the peak at 168.3°C has disappeared. The peak at 168.3°C suggests the existence of an unstable crystal form.

■Analytical Conditions

Instrument : DSC-60
Sample name : Sulfathiazole
Sample weight : 3.88 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 5°C/min

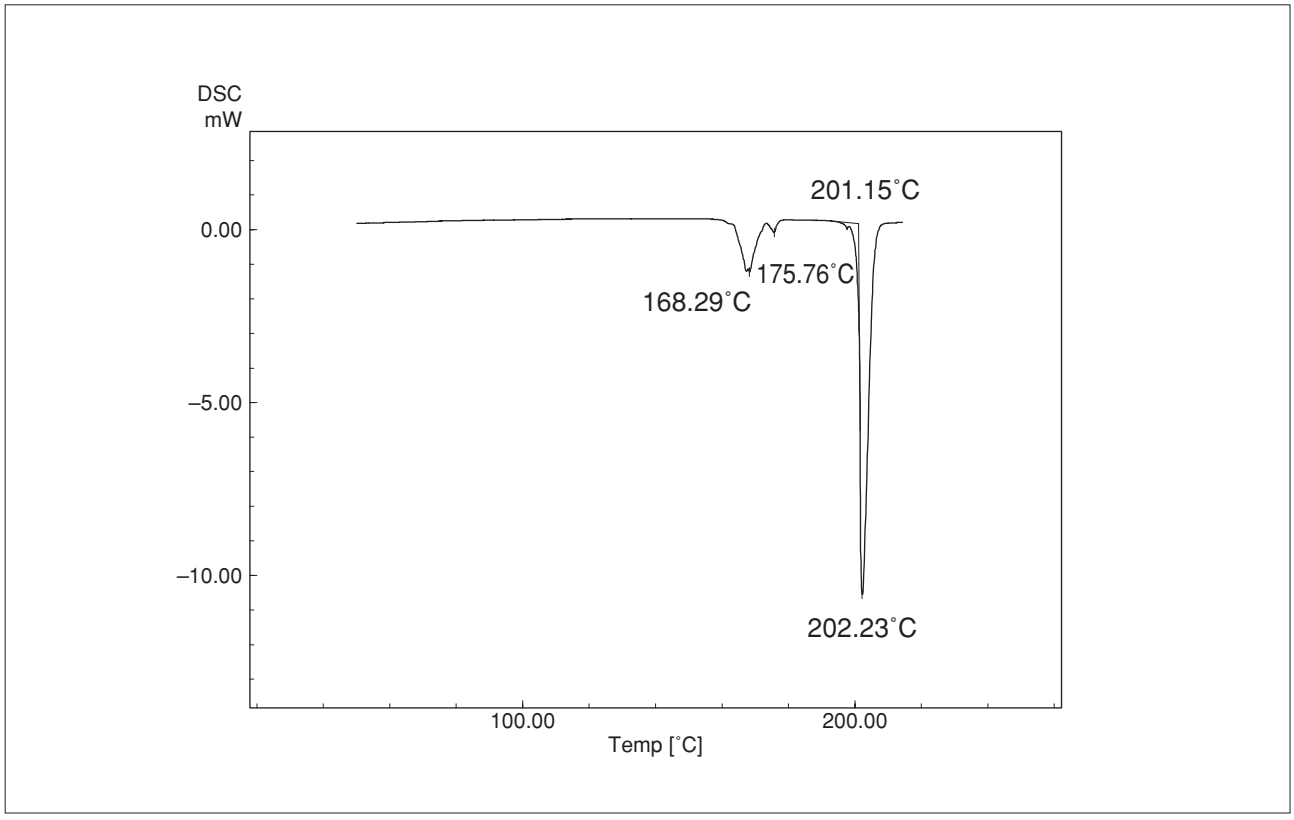

Fig. 11.1.1 DSC curve for sulfathiazole (original)

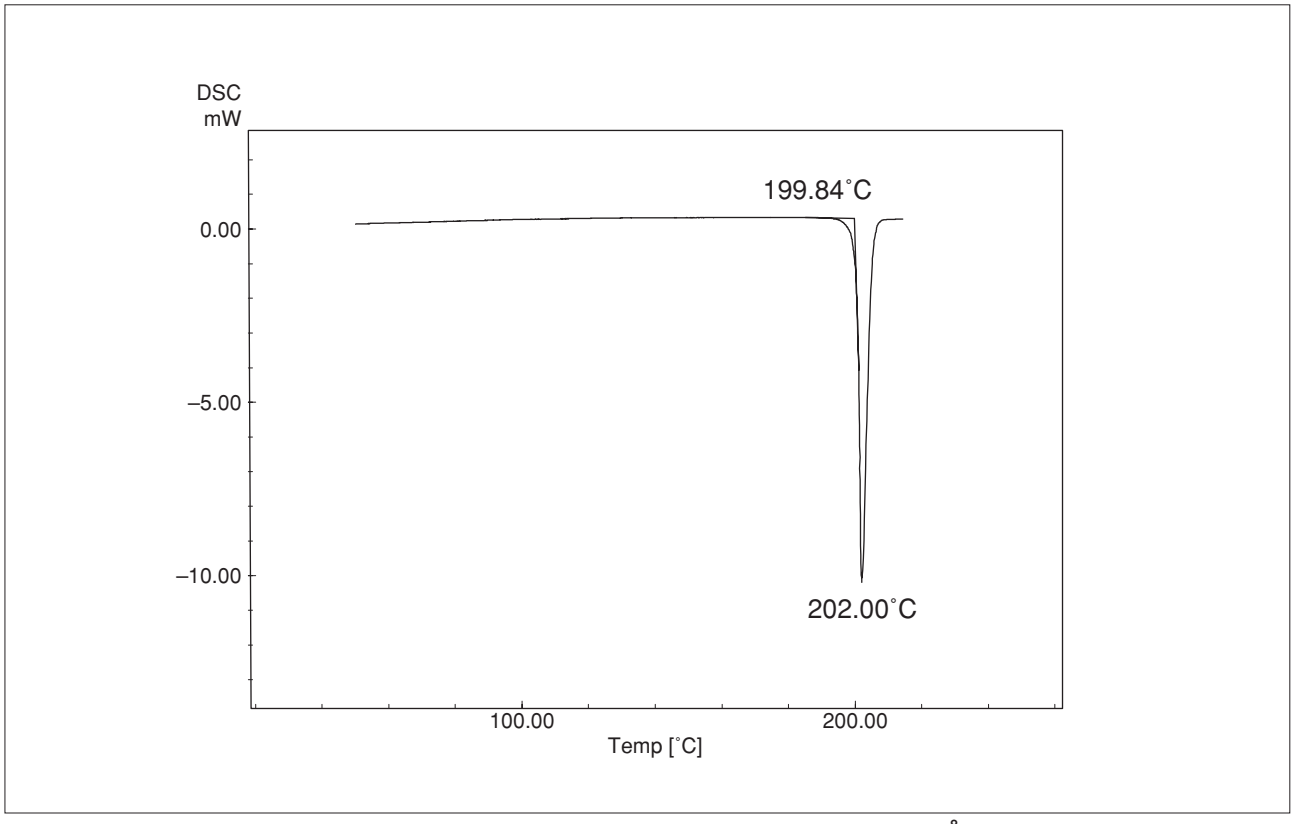

Fig. 11.1.2 DSC curve for sulfathiazole (heat processing up to 185˚C)

11.2 Indomethacin

■Explanation

This example shows the measurement of indomethacin with anti-inflammatory effect. Indomethacin may also show crystal polymorphism. In Fig. 11.2.1, melting is observed at 159.7°C but in Fig. 11.2.2, melting occurs at 156.7°C and there is also a minute endothermic peak at 145.9°C.

■Analytical Conditions

Instrument	: DSC-60
Sample name	: Indomethacin
Sample weight (Fig. 11.2.1)	: 2.97 mg
(Fig. 11.2.2)	3.09 mg
Atmospheric gas	: Nitrogen
Gas flow rate	: 30 mL/min
[Temperature program]	
Heating rate	: 10°C/min

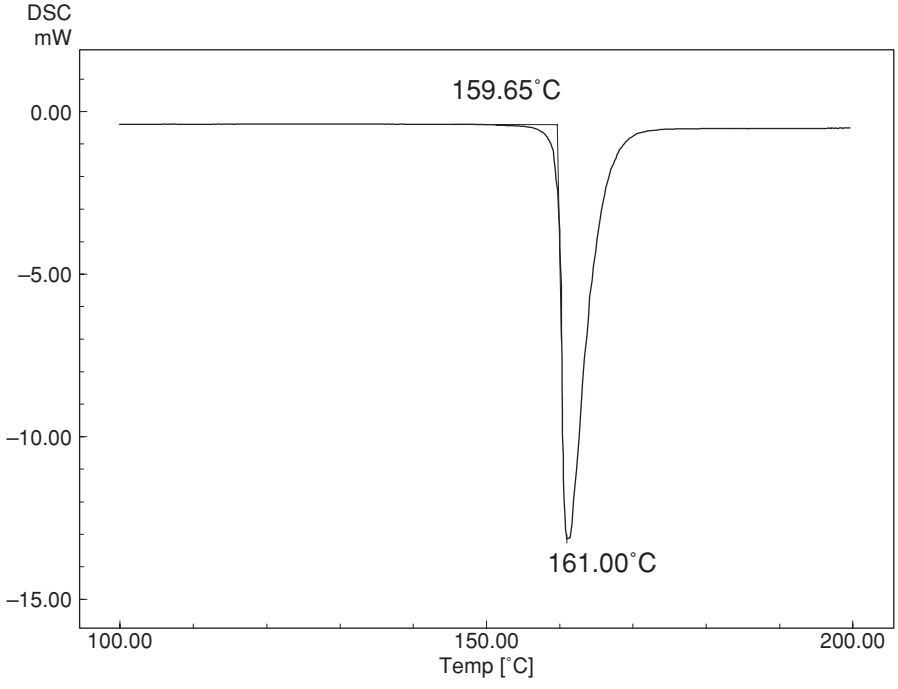

Fig. 11.2.1 DSC curve for indomethacin (No. 1)

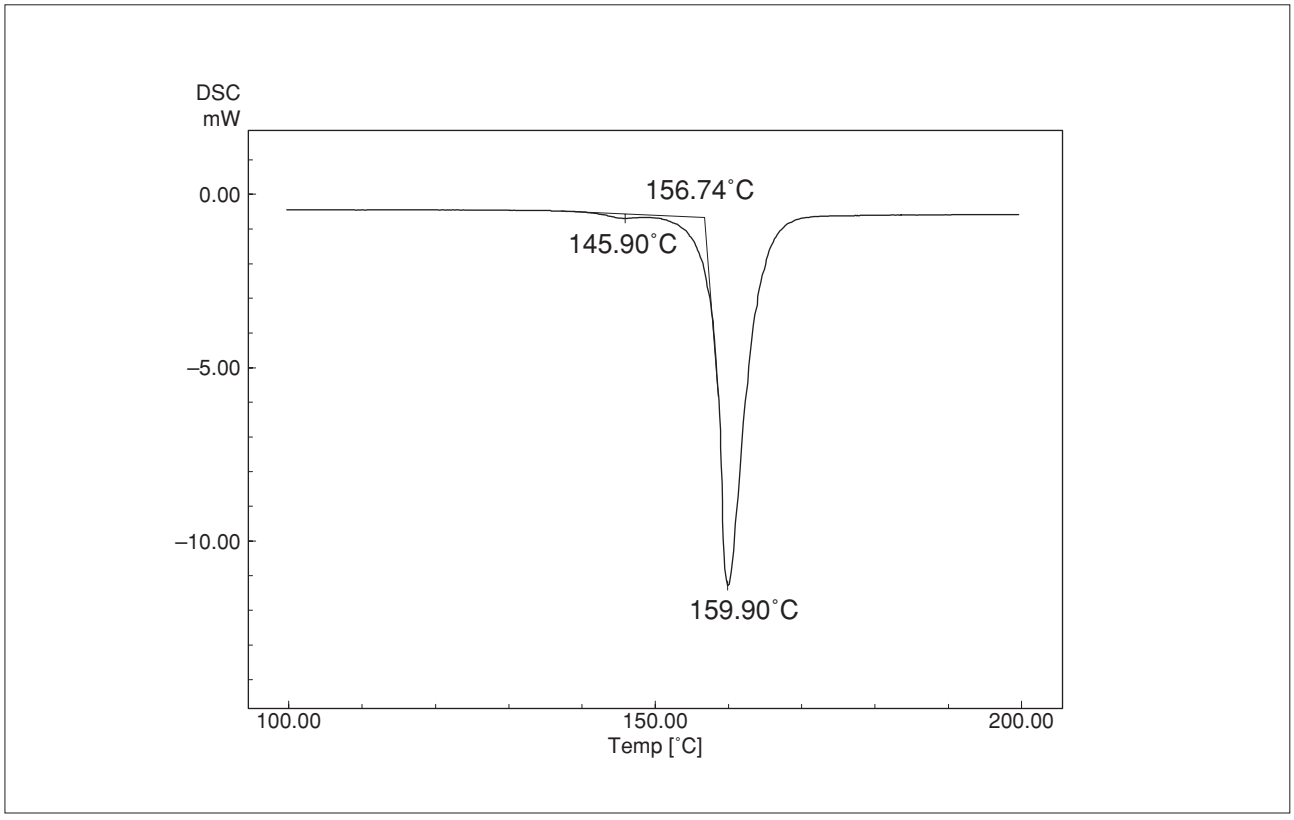

Fig. 11.2.2 DSC curve for indomethacin (No. 2)

11.3 Suppository

■Explanation

The triglycerides used in suppositories exist in several crystal forms and these crystal forms vary with heat processing. A commercial suppository shows a melting peak at 37.6˚C (Fig. 11.3.1). However, when the same suppository is crystallized by cooling after melting, the peak shifts to 32.9˚C (Fig. 11.3.2), showing a significant change in the melting process.

■Analytical Conditions

Instrument : DSC-60
Sample name : Suppository
Sample weight : 8.92 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 3˚C/min

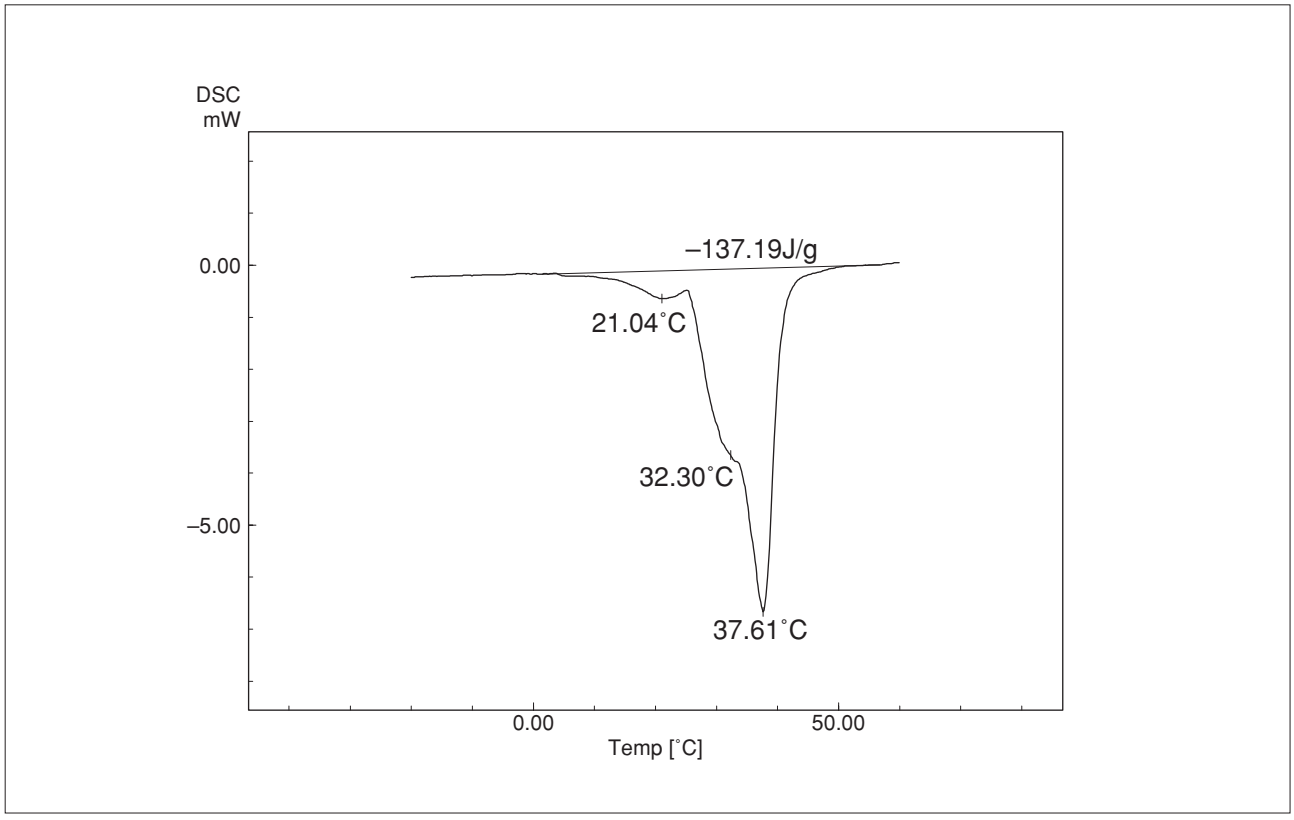

Fig. 11.3.1 DSC curve for commercial suppository

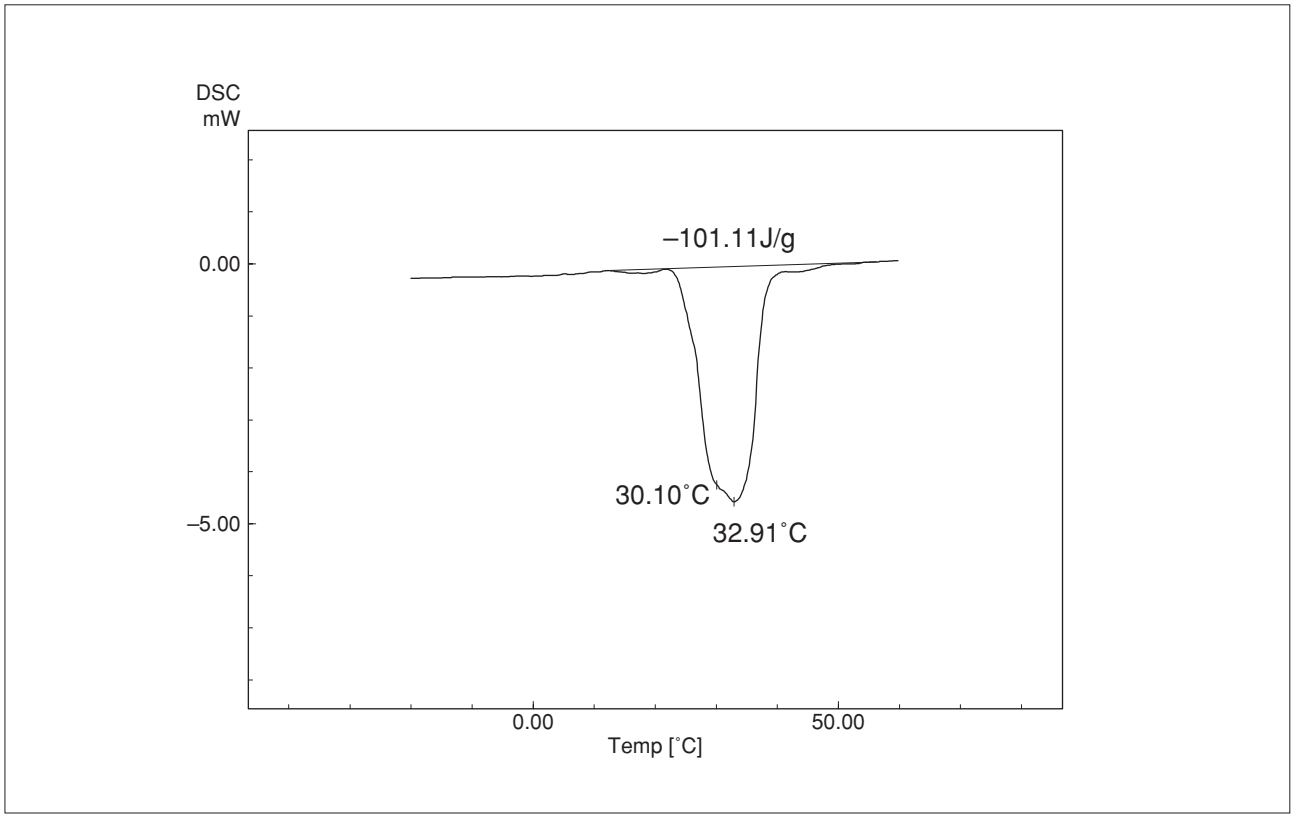

Fig. 11.3.2 DSC curve for commercial suppository after reheating

11.4 Carbamazepine

■Explanation

Carbamazepine is used as an anti-epilepsy drug and is known to exist in multiple crystal forms. This example shows the measurement of the carbamazepine form I (Fig. 11.4.1) and form II (Fig. 11.4.2). A sharp endothermic peak is observed at around 190°C in each one. This corresponds to a melting of form III crystal. In the curve of the form I sample, endothermic and exothermic peaks are observed at between 170°C and 180°C. This likely occurred because form I crystal melted and was recrystallized into form III which is a stable form.

■Analytical Conditions

Instrument	: DSC-60
Sample name	: Carbamazepine I
	Carbamazepine III
Sample weight	: 2.7 mg
Atmospheric gas	: Nitrogen
Gas flow rate	: 50 mL/min
[Temperature program]	
Heating rate	: 5°C/min

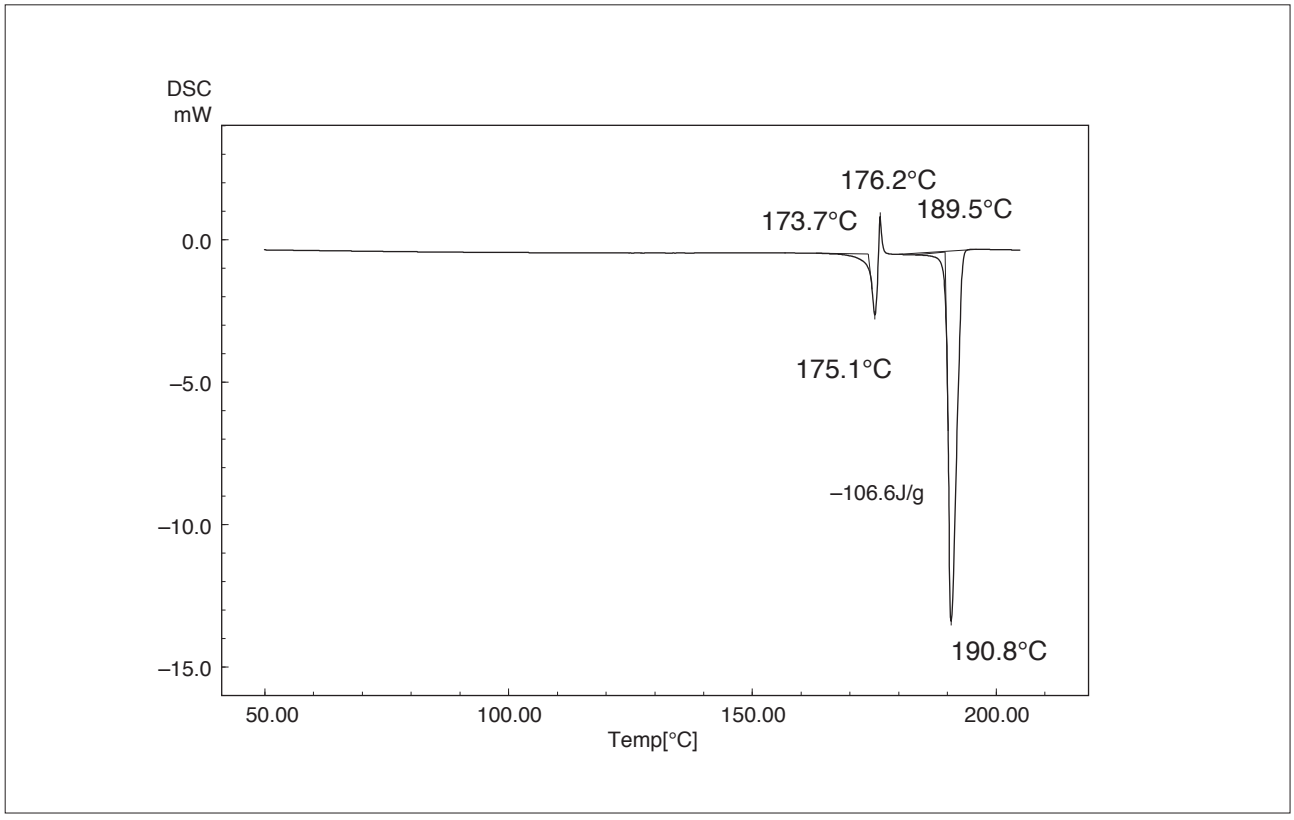

Fig. 11.4.1 DSC curve for carbamazepine I

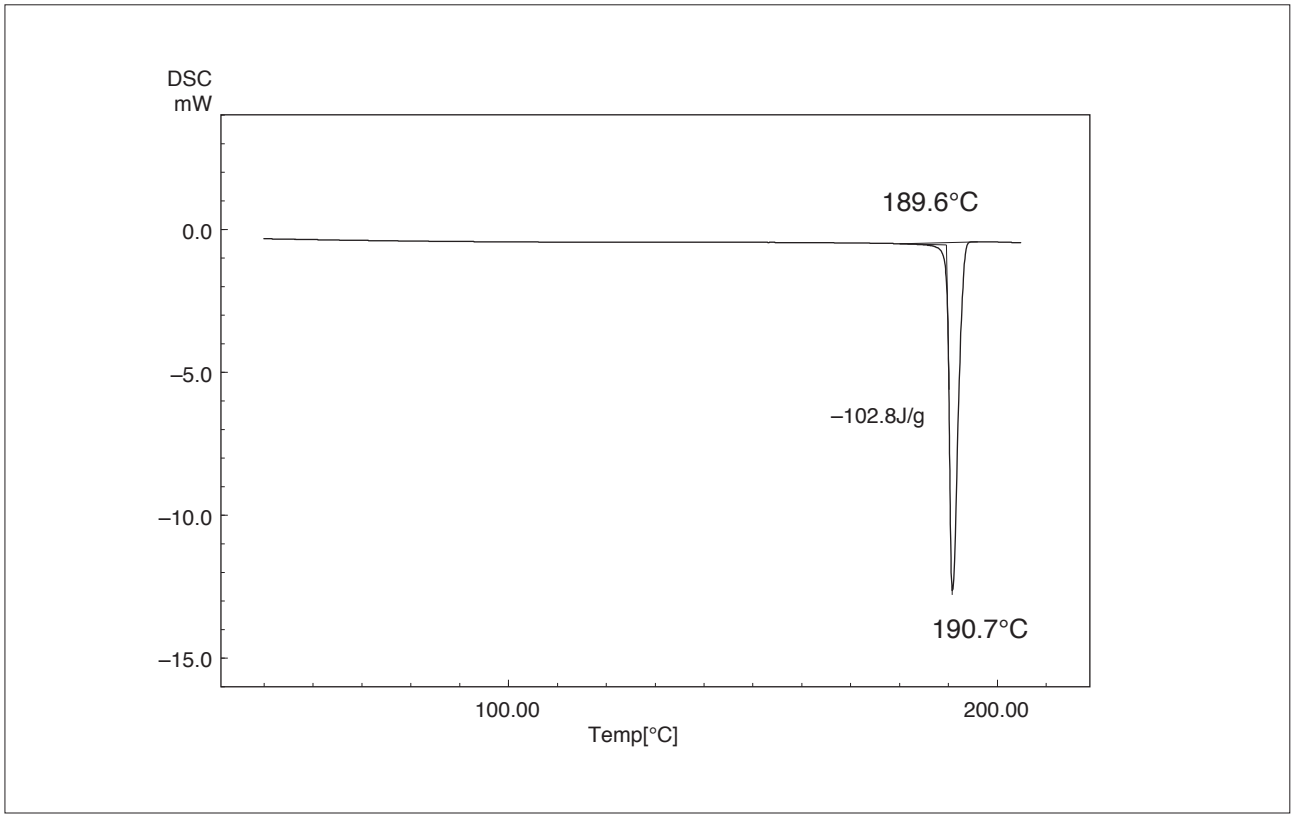

Fig. 11.4.2 DSC curve for carbamazepine III

11.5 Sulfapyridine

■Explanation

Sulfapyridine was heated to 205°C (1st run), cooled, and then reheated (2nd run). In the 1st run, melting of only the stable phase was observed at around 192°C. In the 2nd run, however, glass transition was observed at 53.4°C, crystallization occurred at 101°C, and melting of the metastable phase was observed at 181.9°C.

■Analytical Conditions

Instrument : DSC-60
Sample name : Sulfapyridine
Sample weight : 6.11 mg
Atmospheric gas : Nitrogen
Gas flow rate : 50 mL/min
[Temperature program]
Heating rate : 5°C/min

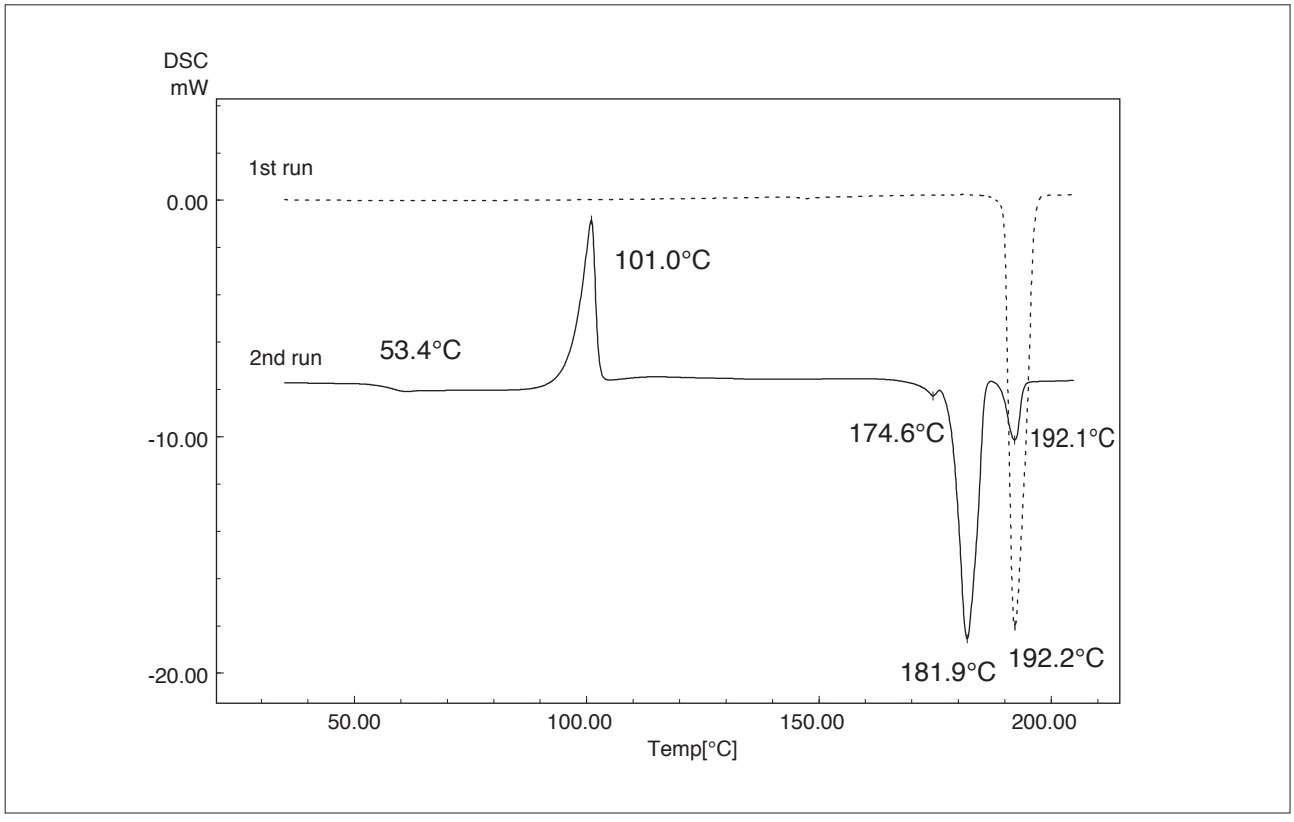

Fig. 11.5.1 DSC curve for sulfapyridine

11.6 Tripalmitin

■Explanation

Tripalmitin is known to have crystal polymorphism. The endothermic peak at 61.6°C observed in the 1st run corresponds to the melting of beta form, which is a stable crystalline form. In the cooling stage, an exothermic peak is observed at 42.5°C. This corresponds to the crystallization of alpha form, which is metastable crystal. Endothermic and exothermic peaks at around 40°C to 58°C in the 2nd run indicate that melting of alpha form and crystallization of beta form occurred simultaneously. Beta form melting heat is low at around 60°C due to its incomplete crystallization.

■Analytical Conditions

Instrument	: DSC-60 and TAC-60i
Sample name	: Tripalmitin
Sample weight	: 1.41 mg
Atmospheric gas	: Nitrogen
Gas flow rate	: 50 mL/min
[Temperature program]	
Heating rate	: 5°C/min
Cooling rate	: 5°C/min

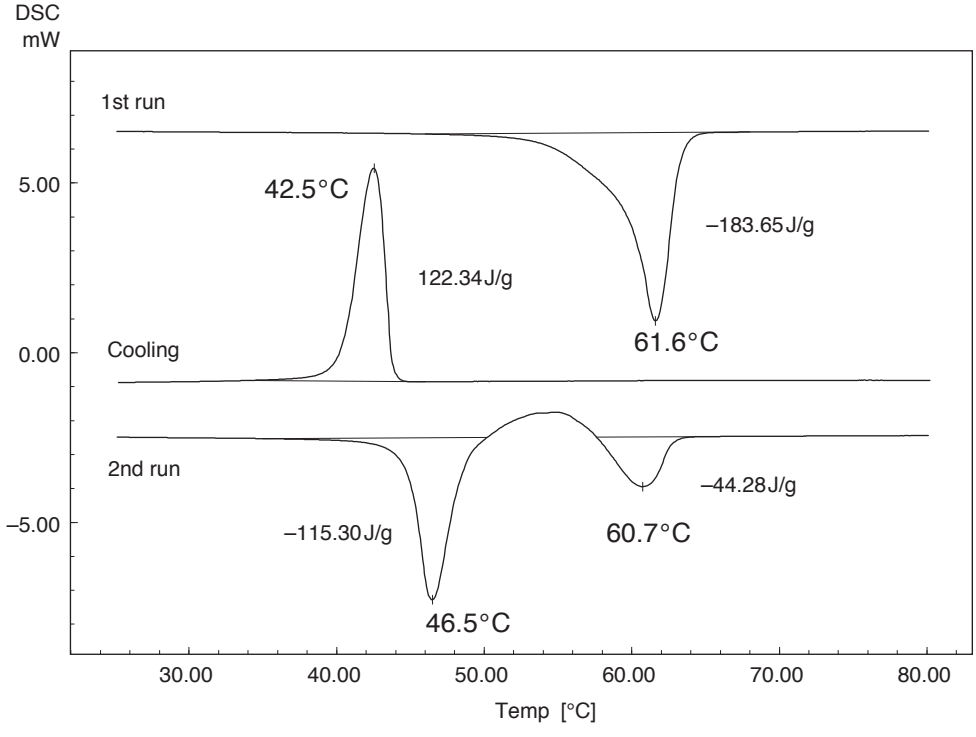

Fig. 11.6.1 DSC curve for tripalmitin

12. Interactions between Pharmaceuticals and Additives

12.1 Benzoic Acid and Magnesium Oxide

■Explanation

Various ingredients are added to pharmaceuticals. DSC allows simple verification of whether or not the ingredients have converted due to interactions. This example shows an examination of interactions between benzoic acid and magnesium oxide. Curve 1 shows the result of measurement of benzoic acid alone. Melting is observed at 122.7°C. Curve 2 shows the result of measurement of magnesium oxide alone and no changes are observed. Curve 3 shows the result of DSC measurement of a 1:1 mixture of benzoic acid and magnesium oxide, mixed mechanically. This shows a completely different pattern from the DSC curves when the two components were analyzed individually. It suggests some kind of interaction between the two substances.

■Analytical Conditions

Instrument : DSC-60
Sample name (curve 1) : Benzoic acid
Sample weight : 4.83 mg
Sample name (curve 2) : Magnesium oxide
Sample weight : 4.38 mg
Sample name (curve 3) : Benzoic acid and magnesium oxide
Sample weight : 2.18 mg
[Temperature program]
Heating rate : 10°C/min
Hold temperature : 180°C

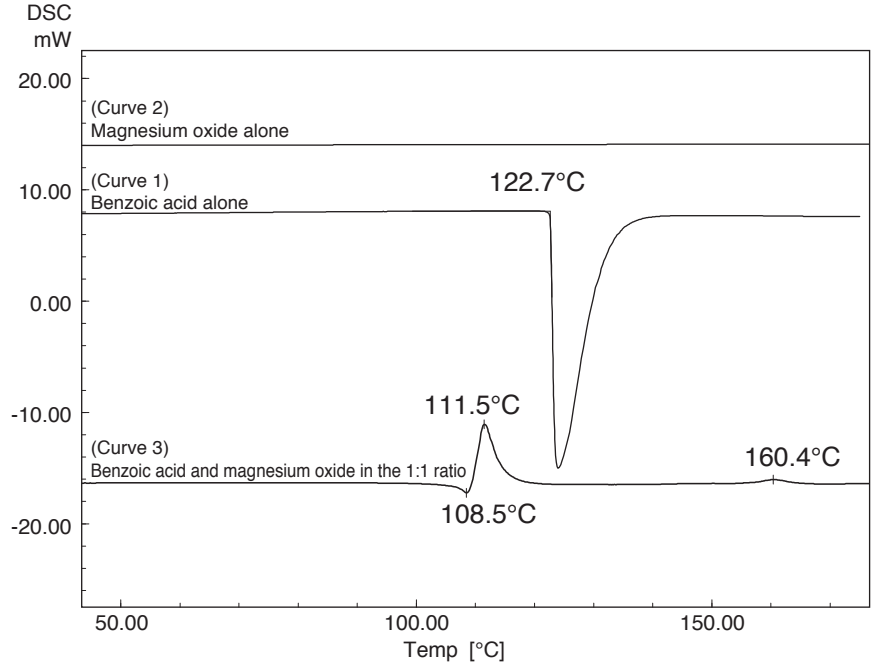

Fig. 12.1.1 Interaction between benzoic acid and magnesium oxide

12.2 Indomethacin and Polyvinylpyrrolidone (PVP)

■Explanation

The lower curve shows the data of indomethacin alone. Melting is observed, which indicates that the substance is crystalline. The upper curve was obtained after 30% PVP was added to indomethacin and the substance was recrystallized from ethanol. No melting peak was observed and glass transition was detected at 26.5°C. This indicates that the substance transformed into non-crystalline form.

Sample provided by Prof. Otsuka, Musashino University

■Analytical Conditions

Instrument	: DSC-60
Sample name	: Indomethacin
Sample weight	: 5.2 mg
Sample name	: Indomethacin + 30% PVP
Sample weight	: 3.1 mg
[Temperature program]	
Heating rate	: 10°C/min
Hold temperature	: 250°C

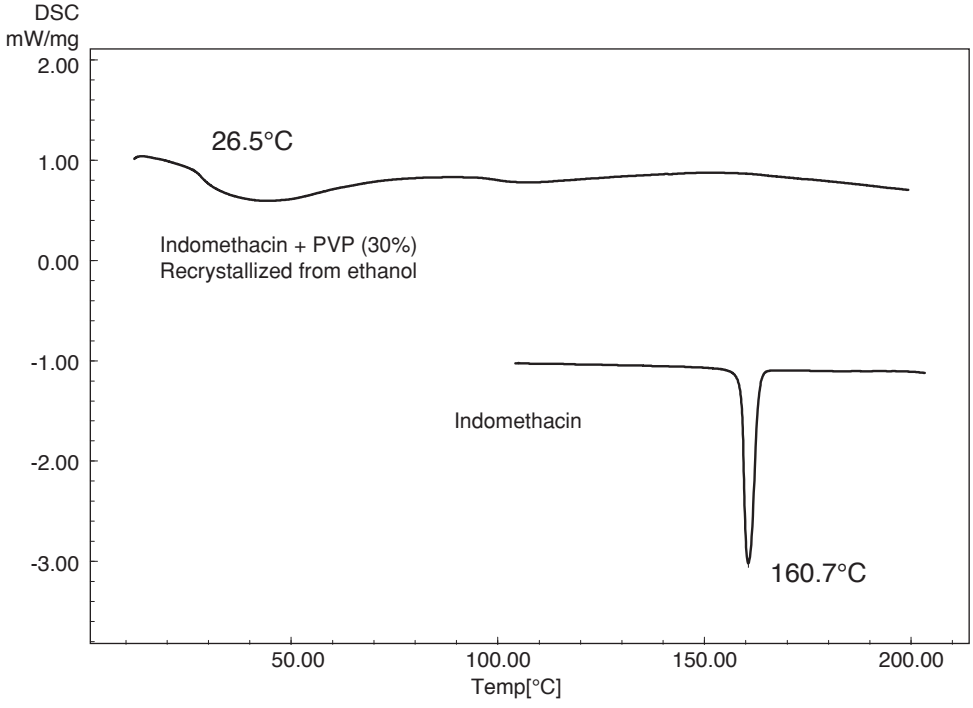

Fig. 12.2.1 DSC curve for indomethacin (+ PVP)

13. Thermostability Testing

13.1 Cyclobarbital

■Explanation

This example shows measurements of cyclobarbital in nitrogen at heating speeds of 3, 5, 7.5, and 10°C/min. The following reaction speed formula is used when performing reaction speed analysis.

$dx/dt = a \exp (-\Delta E/RT) (1-C)^{n-1}$

dx/dt : reaction speed C : reaction rate
a : frequency factor T : absolute temperature
ΔE : activation energy n : reaction order
R : gas constant

Chemical reactions are measured at various heating rates by TGA according to this reaction formula. Plotting the inverse of the temperature (1/T) for a certain weight loss rate and the log of the heating rate (LOG A) gives a linear curve (Ozawa plot) and the activation energy (ΔE) is obtained from its slope. An activation energy of 109.2KJ/mol was obtained in this example. When reaction speed parameters such as the frequency factor and reaction order are calculated, isothermal analysis becomes possible and the reaction rate at different temperatures and required reaction times can be obtained. This test gives the prediction that, if kept at 30°C, the decomposition will have progressed to 20% in 3.69×10^6 hours (about 421 years).

■Analytical Conditions

Instrument : TGA-50
Used software : Kinetics Analysis Program for TGA
Sample name : Cyclobarbital
Sample weight : 9.187 mg
Atmospheric gas : Nitrogen
Gas flow rate : 30 mL/min
[Temperature program]
Heating rate : 3, 5, 7.5, 10°C/min

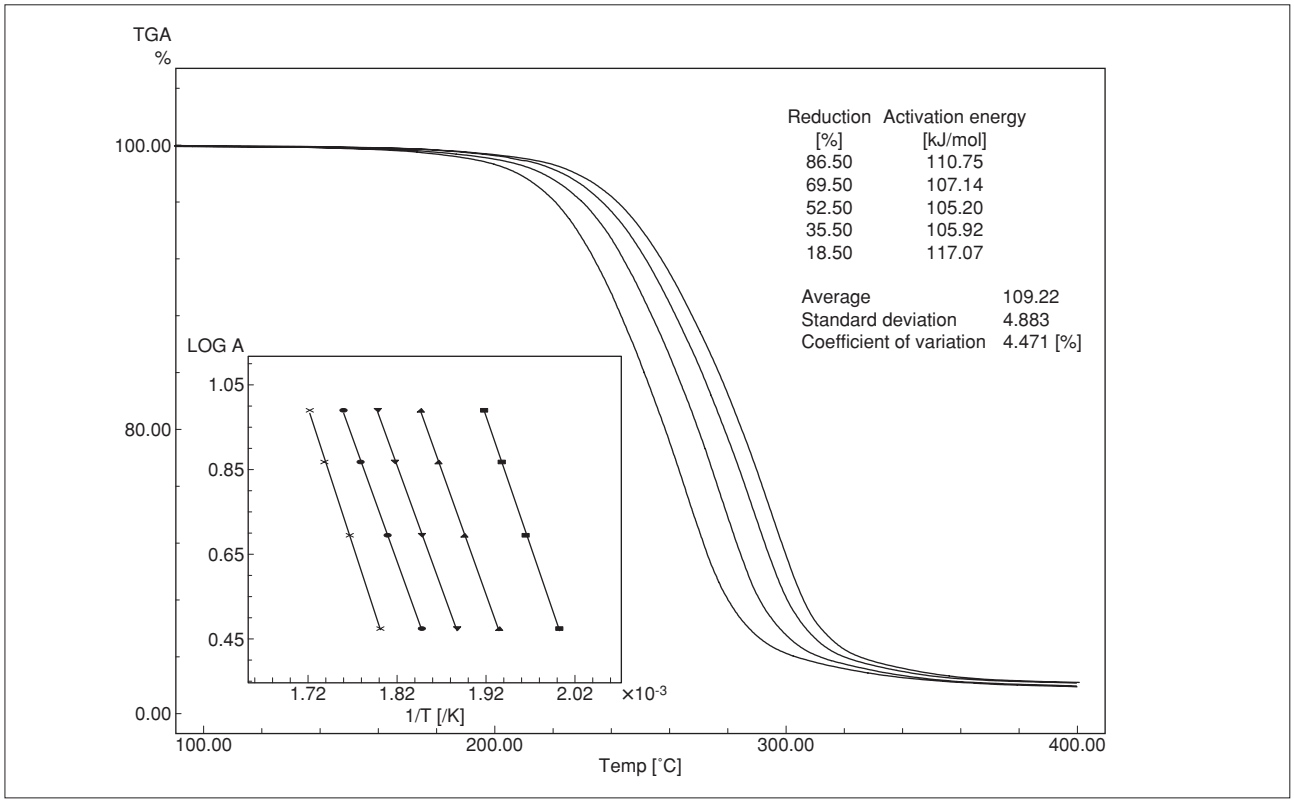

Fig. 13.1.1 Analysis of cyclobarbital thermal decomposition

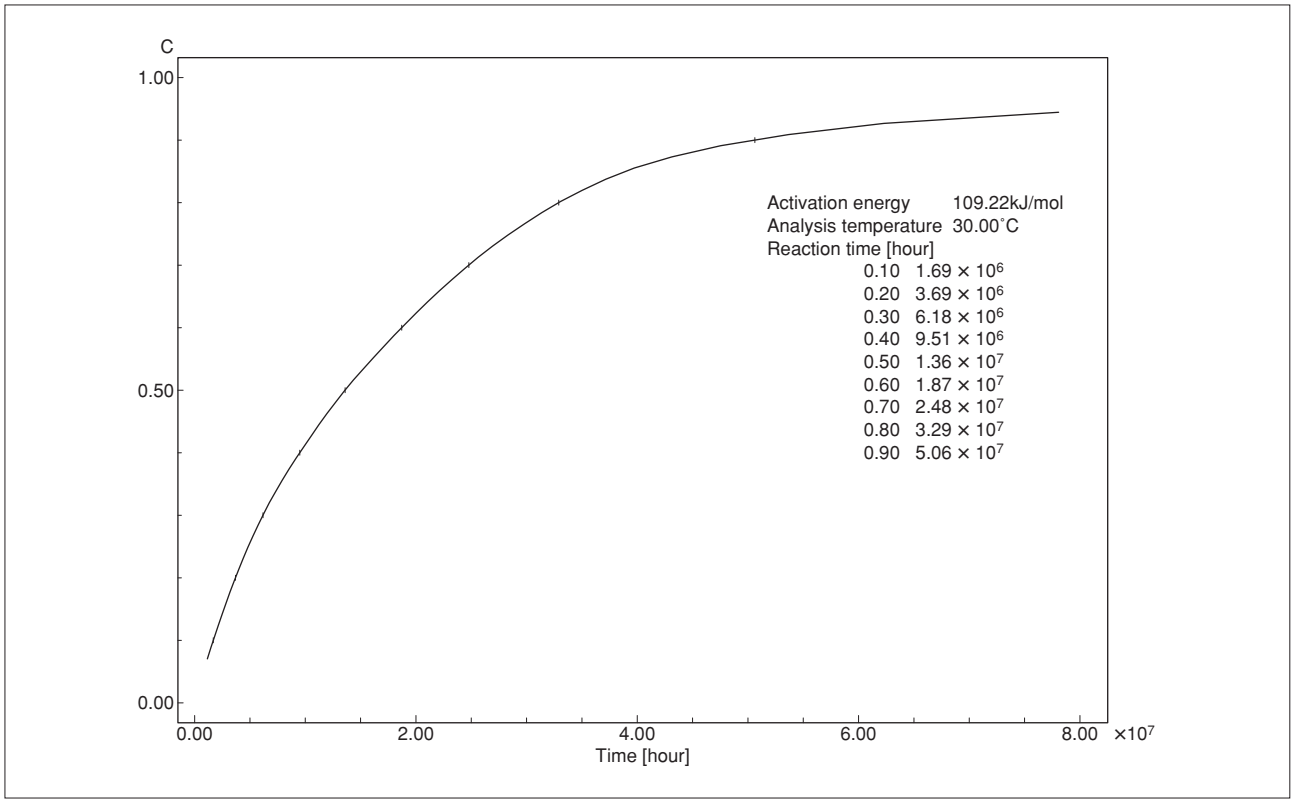

Fig. 13.1.2 Inference of reaction time for cyclobarbital kept at 30˚C

14. TG-DTA Measurement

14.1 Sodium Tartrate

■Explanation

Sodium tartrate dehydrate was measured by DTG. From the calculation of molecular weights, the 15.7% weight loss observed between 45°C and 170°C is supposed to be the desorption of two molecules of water. TG shows 2-stage weight loss. The first loss (desorption of the first molecule) occurs slowly and the second loss starts before the completion of the first reaction. DTA shows endothermic peaks at 74.5°C and 137°C, which correspond to the desorption of two water molecules. Note that the weight loss and endothermic and exothermic peaks starting at around 240°C are due to decomposition.

■Analytical Conditions

Instrument : DTG-60
Sample name : Sodium tartrate
Sample weight : 12.18 mg
Atmospheric gas : Air
Gas flow rate : 50 mL/min
[Temperature program]
Heating rate : 10°C/min

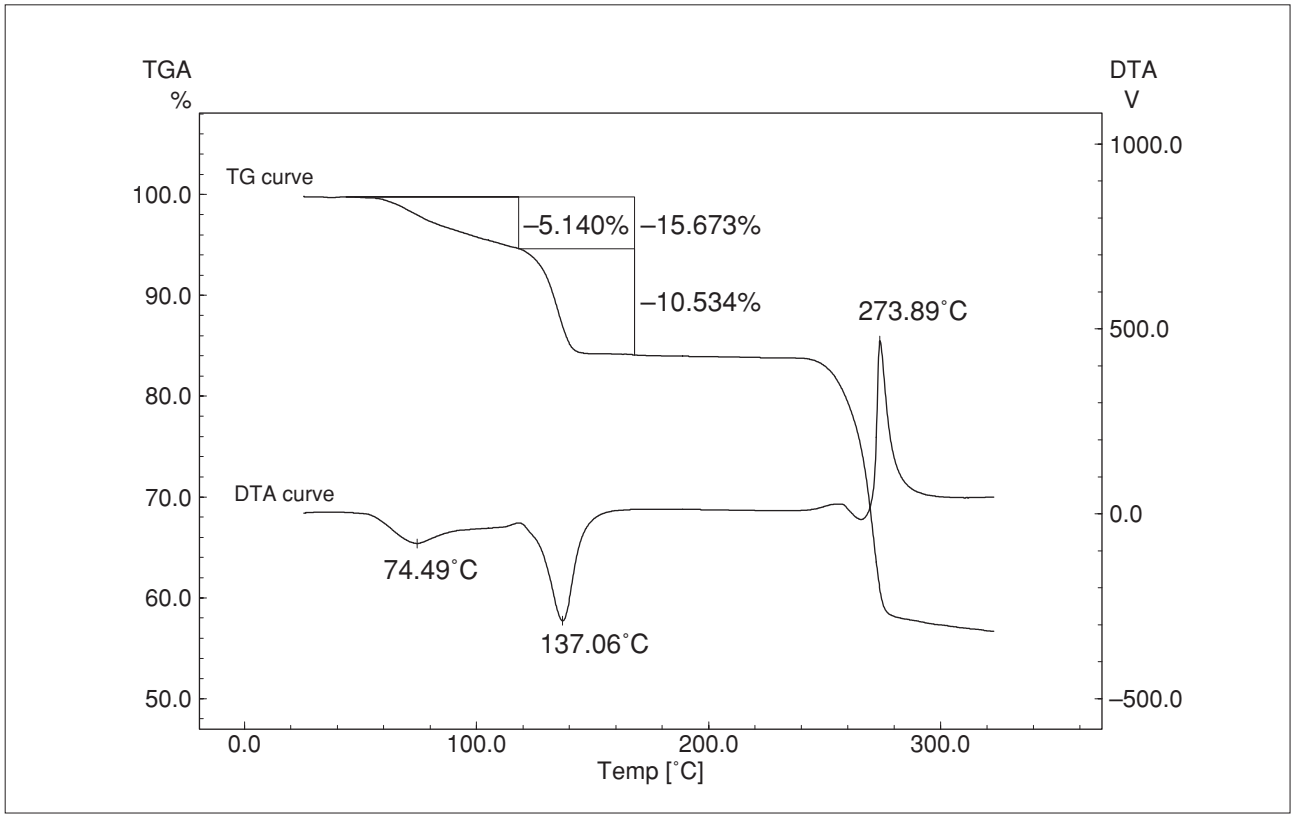

Fig. 14.1.1 TG-DTA curve for sodium tartrate

14.2 Cyclodextrin

■Explanation

Cyclodextrin was measured with DTG. Decomposition starts at around 250˚C. The DTA curve shows complex peak patterns because the endothermic and exothermic reactions occur simultaneously. Cyclodextrin has a ring structure and takes in water in the form of inclusion water. The release of the inclusion water is observed at around 81˚C.

■Analytical Conditions

Instrument : DTG-60
Sample name : Cyclodextrin
Sample weight : 10.82 mg
Atmospheric gas : Air
Gas flow rate : 50 mL/min
[Temperature program]
Heating rate : 10˚C/min

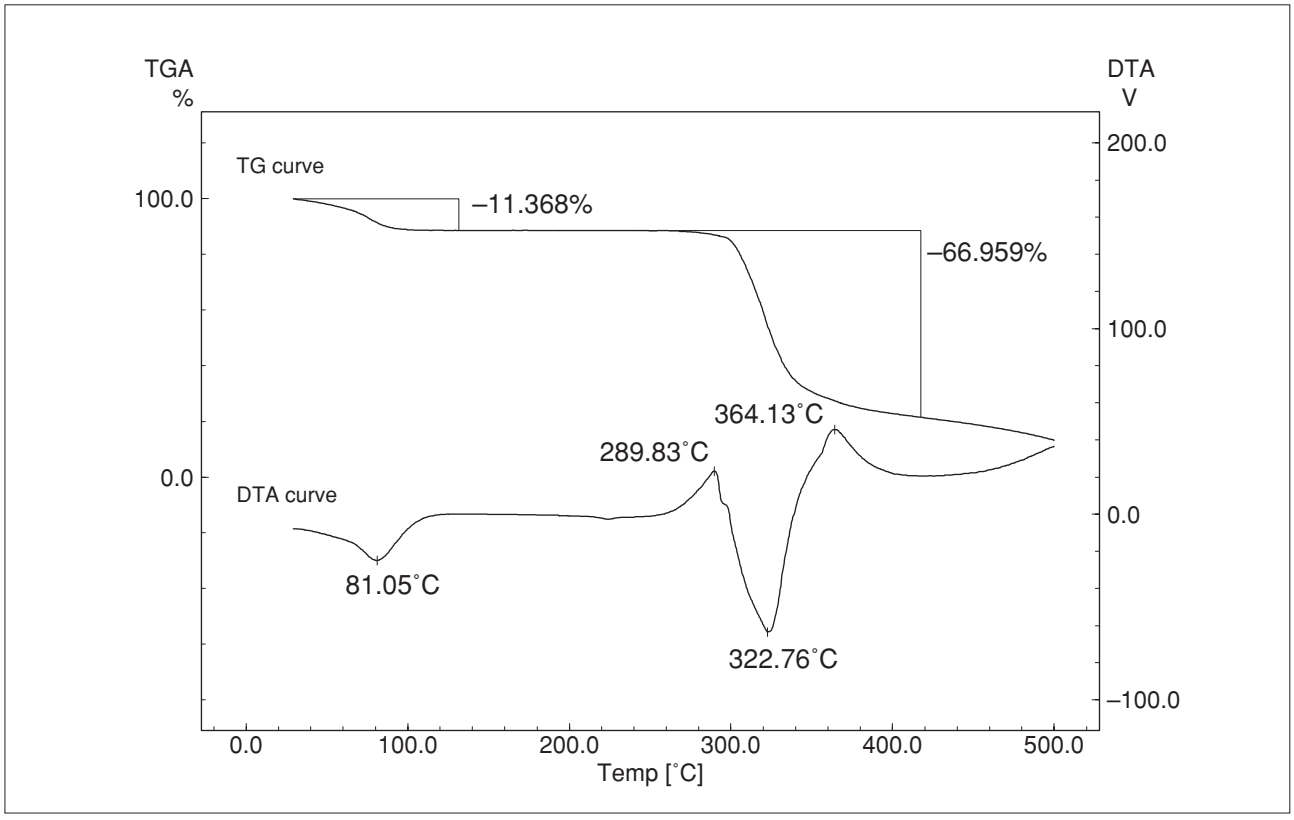

Fig. 14.2.1 TG-DTA curve for cyclodextrin

14. TG-DTA Measurement

14.3 Aspartame

■Explanation

The artificial sweetener aspartame was analyzed by TG-DTA. In the TG curve, three-stage weight loss is observed. In the DTA curve, three endothermic peaks are observed. The weight loss at around 50°C is considered to be caused by the evaporation of free water. The weight loss accompanied by an endothermic reaction at around 100°C is considered to be caused by the detachment of crystalline water. Decomposition occurred at around 180°C.

■Analytical Conditions

Instrument	: DTG-60
Sample name	: Aspartame
Sample weight	: 25.07 mg
Atmospheric gas	: Nitrogen
Gas flow rate	: 50 mL/min
[Temperature program]	
Heating rate	: 3°C/min

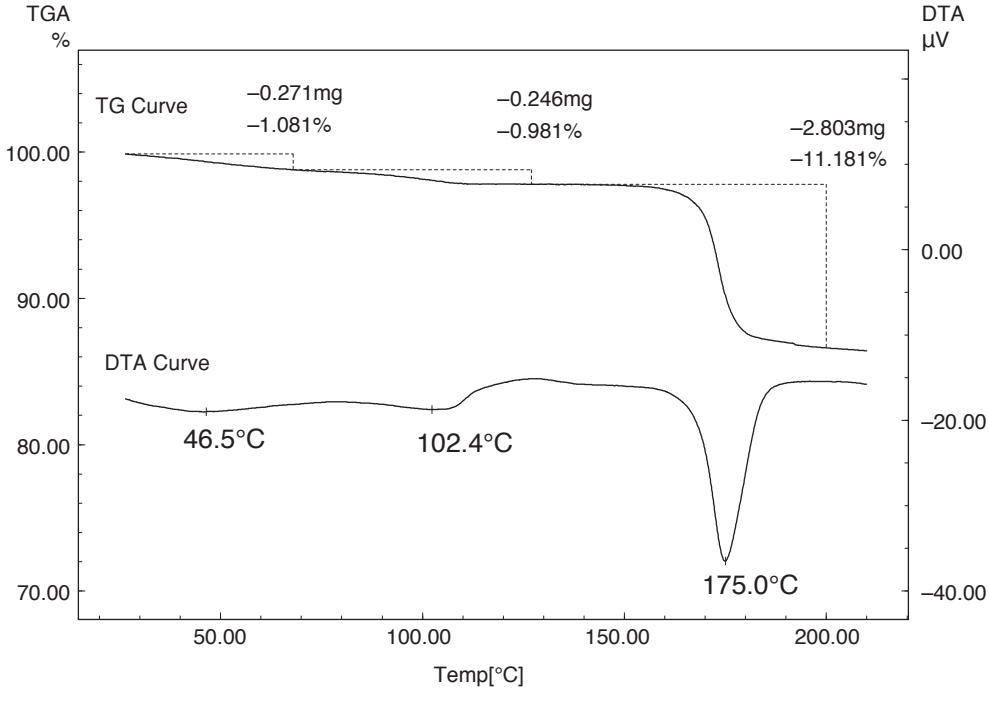

Fig. 14.3.1 TG-DTA curve for aspartame

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