Thermoplus EVO3 DSCvesta2

—DSC with a newly designed thermal sensing plate—



1. Introduction

Thermal analyzers are widely used in various material fields. Specifically, the DSC (Differential Scanning Calorimeter) is an essential tool for investigating glass-transition temperature and melting points of polymers and pharmaceuticals. In 2017, Rigaku released DSCvesta®, which had a higher sensitivity, better stability, and wider measurement-temperature range than earlier models. Recently, Rigaku has developed its upper model, DSCvesta2, with a newly designed thermal sensing plate to improve the DSC performance even further.

2. Features

2.1. Newly developed χ sensor*

The newly developed DSC sensor, " χ sensor." (Fig. 1), has a thermal sensing plate with a redesigned shape and structure to improve the sensitivity and baseline reproducibility. Four claw guides are located around the outer circumference of the χ sensor to prevent misalignment of the pans, improving measurement reproducibility and helping the users place the sample pans in the correct positions more easily.



Fig. 1. χ sensor

The resistance to deterioration of the sensor has also been increased. This embossed-shaped sensor allows measurements in an oxidative atmosphere up to 600°C.

2.2. Self-diagnostic function, vestaeye®

The DSCvesta2 has a completely new electrical circuit. A built-in self-diagnostic function, "vestaeye," can constantly monitor the instrument's status in both standby and measurement modes. The module monitor on the measurement screen always displays vestaeye results to detect failures and defects, allowing the user to check the instrument status even before a measurement. This contrasts the earlier models, with which such status was available only in the measurement mode.

2.3. ΔBlock[®] with an improved measurement temperature range

The DSCvesta2, with an improved refrigerated cooling unit, can cool the sample down to -95° C. This enables calibration at low temperatures using cyclohexane. With a liquid nitrogen direct cooling unit (Fig. 2), the sample



Fig. 2. Liquid nitrogen direct cooling unit

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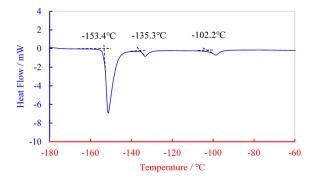


Fig. 3. DSC results of cyclopentane using a liquid nitrogen direct cooling unit

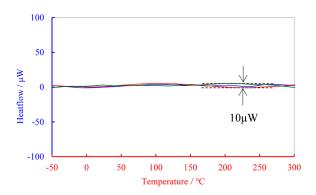


Fig. 4. Drift reproducibility

can be cooled down to -180° C. The operator can easily conduct measurements from -180° C by pouring liquid nitrogen into the tank in front of the electric furnace.

DSC results of cyclopentane are shown in Fig. 3. The experiment was conducted at a heating rate of 20°C/min from -180°C using a liquid nitrogen direct cooling unit. An endothermic peak associated with a transition at -150°C is measured. This demonstrates the advantage of the cooling capability down to -180°C .

3. Basic performance of DSCvesta2

The DSCvesta2 has an entirely new sensor with the ΔBlock which is decisive for its DSC performance. The new sensor also features an electrical circuit with an ultra-low noise amplifier developed by Rigaku and improved signal processing algorithms.

Figure 4 shows the drift reproducibility from -50° C to 300°C at a heating rate of 10°C/min. The results showed a reproducibility within 10μ W. A comparison of the Indium melting results of DSCvesta and DSCvesta2 is exhibited in Fig. 5. The sample amount is 1 mg, and the heating rate is 20°C/min. Compared with DSCvesta, DSCvesta2 gives a higher peak with a narrower full width at half maximum. This suggests that DSCvesta2 has a better sensitivity and separation resolution.

4. Application

4.1. Specific heat capacity measurement

In addition to measuring the thermal behavior of a sample, DSC is also used to determine the specific heat capacity. Figure 6 shows the results of specific heat

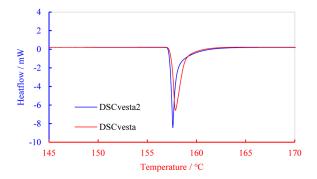


Fig. 5. Comparison of melting peaks of In

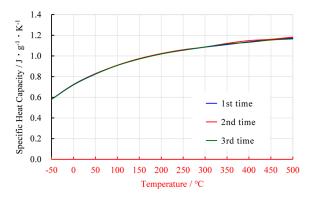


Fig. 6. Reproducibility of specific heat capacity of sapphire by DSCvesta2

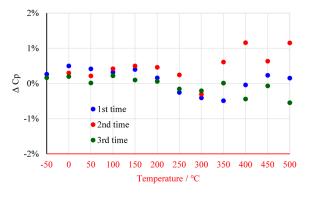


Fig. 7. Difference of specific heat capacity between measurement and literature values (NIST) with temperature variations

capacity measurements of a sapphire performed by DSCvesta2. The measurements were made in one step of the temperature program from -50° C to 500° C at a constant heating rate. To evaluate the reproducibility, three sample measurements were performed. The sample was unloaded after each measurement to resemble an actual measurement.

When measuring the specific heat capacity of a sample using a DSC, one of the most important factors is the reproducibility of instrument drift. To obtain reproducible results with a conventional DSC, it was necessary to set a temperature holding step for each temperature range of 200°C to 300°C when setting the measurement temperature program. DSCvesta2,

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however, has an improved drift reproducibility (Fig. 4), so that specific heat capacity can be calculated with good reproducibility in the temperature range from -50°C to 500°C without a temperature holding (Fig. 6). Specific heat capacity values were calculated from the results of the three measurements. Their percent deviations from literature values were plotted (Fig. 7). It is seen that the reproducibility was less than 2% for each measurement.

5. Summary

DSCvesta2 has been developed to be a reliable daily tool with improved data reproducibility thanks to χ sensor and self-diagnostic function of vestaeye. In particular, vestaeye is a completely new feature that has

not been available in conventional thermal analyzers. The self-diagnostic function, vestaeye, examines for abnormalities every time the power is turned on. This allows the analyzer to be used stably over its long lifetime, sometimes extending to 10 years or more. This long-time stability makes DSCvesta2 suitable not only for research and development purposes but also for quality control applications.

The DSCvesta2 offers as many types of optional attachments as the conventional models, e.g., an automatic sample changer, sample observation unit, UV irradiation unit, and temperature modulation unit.

Try out DSCvesta2, with its excellent performance and extraordinary versatility while maintaining the same scalability as before.

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