Thermogravimetry/Differential Thermal Analyzer STA7200RV

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1. Introduction

Hitachi High-Tech Science Corporation develops, manufactures, and markets various thermal analyzers termed DSC, TG/DTA, TMA, and DMA. One characteristic feature of our thermal analyzers is their compatibility with a specimen observation function. The "specimen observation function" provides linked, simultaneous acquisition of thermal analysis data and specimen observation images, facilitates data interpretation, and supports discovery of new knowledge.

The TG/DTA7220 system released in 2012 provides a function allowing TG/DTA measurement and simultaneous CCD camera observation of specimen changes during measurement at temperatures up to 500°C. For example, the system allows real-time observation of weight reduction and concomitant emission of pyrolysis gas caused by thermal decomposition, together with phenomena such as changes in specimen color.

To meet changing market needs, we have now developed the STA7200RV (Fig. 1), which increases the measurement temperature range of the system, allowing specimen observation at temperatures up to 1000°C. This system allows specimen observation at previously unavailable temperature ranges, for example, those in the complete decomposition process of high molecular organic materials, and glass transition and fusion of metals, glasses, and other inorganic materials.

Fig. 1 External appearance of STA7200RV Simultaneous Thermogravimetric Analyzer

2. Role of the Simultaneous Thermogravimetric Analyzer

The Simultaneous Thermogravimetric Analyzer ("TG/DTA" below) changes the temperature of a specimen according to a predetermined program while simultaneously measuring sample mass as a function of temperature and acquiring a differential thermal signal.

The TG/DTA is used to evaluate the heat resistance of inorganic, organic, polymer, and various other materials in various ways. Specific examples of heat-resistance evaluation include measurement of pyrolysis temperature or dehydration temperature, and quantification of decomposition, attached water, or crystallization water, under various conditions.

The TG/DTA systems are also used with rubber materials and copy toners for component analysis or carbon black quantification. They are also used to determine the moisture content of foods and drugs, designate drying parameters for these items, evaluate thermal stability, and measure oxidation reactions of metals. Reaction kinetics analysis are also carried out frequently using TG curves.

3. Features of the STA7200RV

3-1 Heating furnace equipped with specimen observation window (viewport)

Previous TG/DTA analyzers enclosed specimens in the heating furnace, preventing observation of sample status. Phenomena occurring in the sample could only be assessed through TG signals (changes in mass) and DTA signals (differential thermal changes), and when assessment was difficult, phenomena could only be inferred. The STA7200RV includes a new quartz glass viewport in the heating furnace, allowing specimen observation during measurement (Fig. 2).

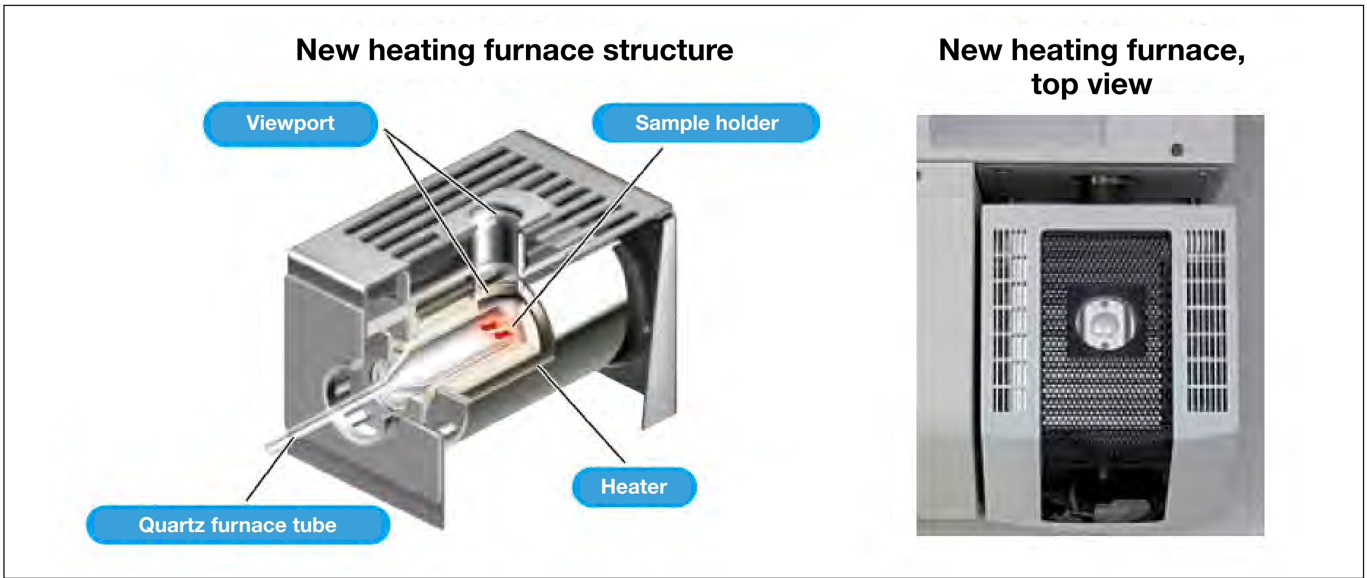

Figure 2 Structure of new STA7200RV heating furnace

The quartz glass construction of the furnace tube provided in the heating furnace to control the gas atmosphere has also been designed to allow observation of a specimen in the vessel with a CCD camera mounted directly above. The structure is compatible with specimen observation at temperatures up to 1000°C (patent pending). However, a weak point of the quartz glass is progressive devitrification by pyrolysis gases produced during TG/DTA measurement, which can render specimen observation indistinct.

Consequently, the STA7200RV is designed for user maintenance, with a heating furnace structure employing a user-replaceable furnace tube to allow acquisition of distinct specimen observation data at all times.

3-2 Stability of the DTA signal

The DTA provides a signal capturing the temperature difference versus a standard substance, which represents heat absorbed and emitted as a specimen changes. In TG/DTA measurement, heating changes specimen features such as shape, surface state, and color, which changes the emissivity of the specimen itself and can affect a differential thermal signal. The shape of the STA7200RV viewport is designed not only to allow specimen observation, but also to reduce the effect of emissions from directly above the specimen, which helps to improve DTA signal stability, as shown in Fig. 3.

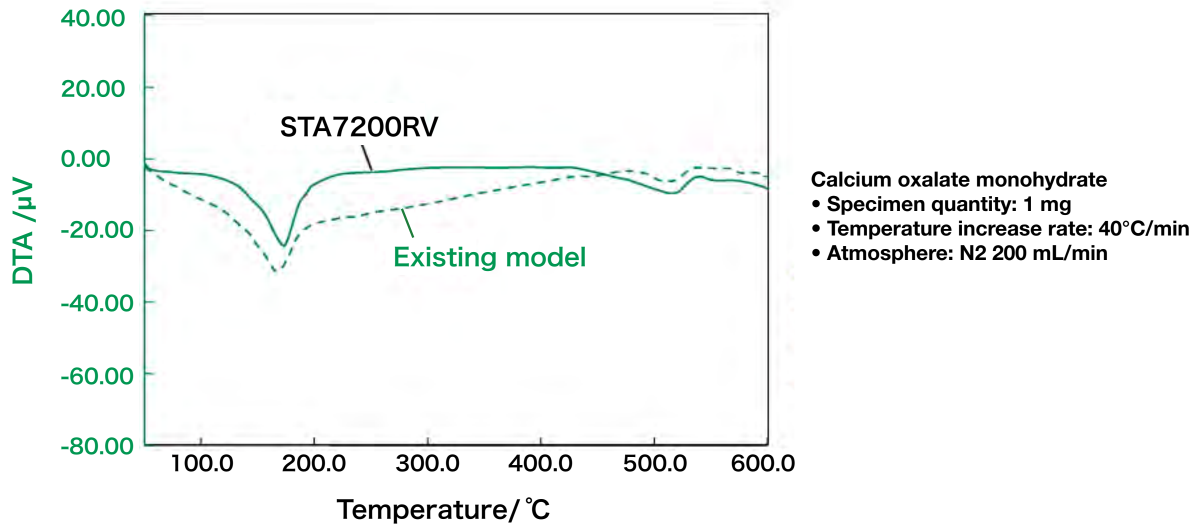

Fig. 3 DTA signal stability data

3-3 Analysis software

The analysis software has also been made compatible with the specimen observation function (Fig. 4). When saved measurement data are loaded, individual measurement signals and observational images are automatically linked and displayed, allowing easy comprehension in data analysis.

Two different display functions are provided. Slide display (Fig. 4(a)) is a quasi-animated replay mode for observed images that allows easy, intuitive understanding of sample changes. Thumbnail display on the other side (Fig. 4(b)) allows checking of measurement data with reference to specimen images in the process and is suited to detailed analysis.

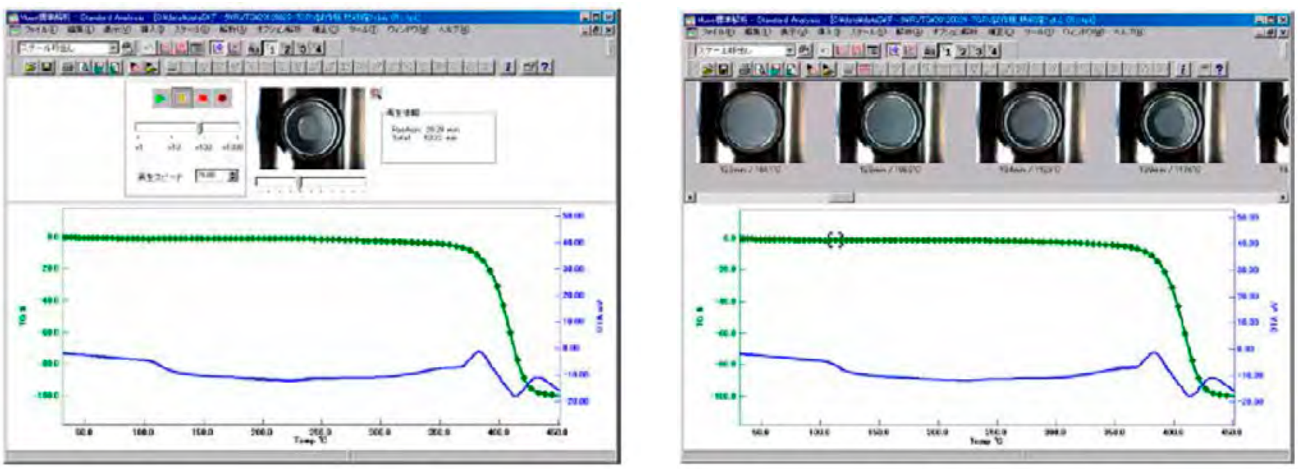

Fig. 4 (a) Slide display **Fig. 4 (b) Thumbnail display**

Fig. 4 Analysis software

4. Measurement Example of TG/DTA Specimen Observation

As a measurement example of the TG/DTA specimen observation, Fig. 5 shows measurement data for pharmaceutical ursodeoxycholic acid. The specimen is a drug used for gallstones and liver disease which protects the liver by improving bile flow. Crystallization occurs near 120°C, at , where the DTA shows an exothermic peak. The specimen profile demonstrates little change. Near 200°C, melting occurs, which the specimen profile also confirms.

Weight reduction begins immediately after melting, with the specimen also changing color to brown and beginning to show degradation and decomposition. At 300°C and above, secondary decomposition occurs, and finally at 500°C and above, attainment of 100% decomposition is clearly evident.

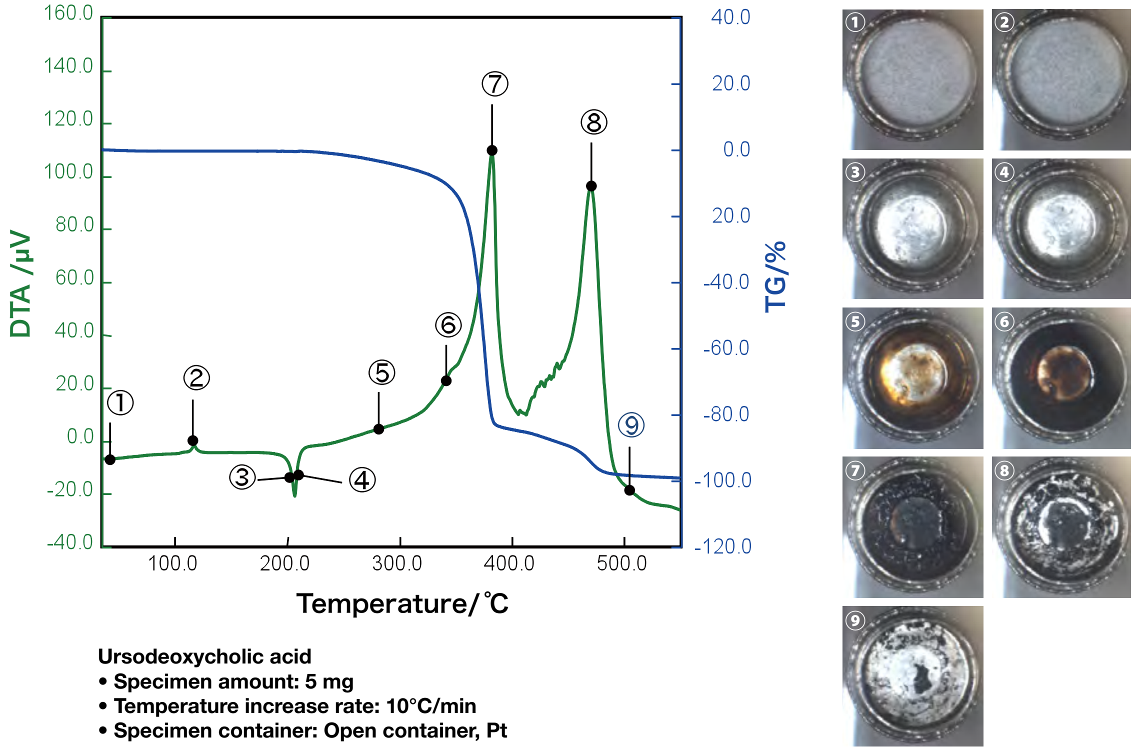

Fig. 5 Measurement data for ursodeoxycholic acid

5. Closing remarks

The capability of the STA7200RV for direct specimen observation adds information to thermal analysis results, for example, changes in specimen shape and color. New advantages include: ease of data analysis, capture of information on previously unrecognized phenomena, and causal analysis of phenomena that are considered abnormal. On this basis, we anticipate adoption for an even broader range of applications.

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