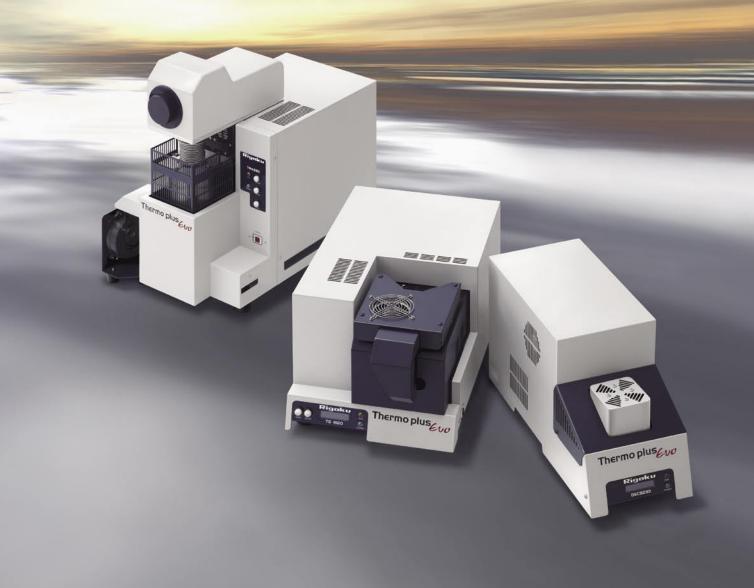


# Thermo plus & Vo II new Thermal analysis system

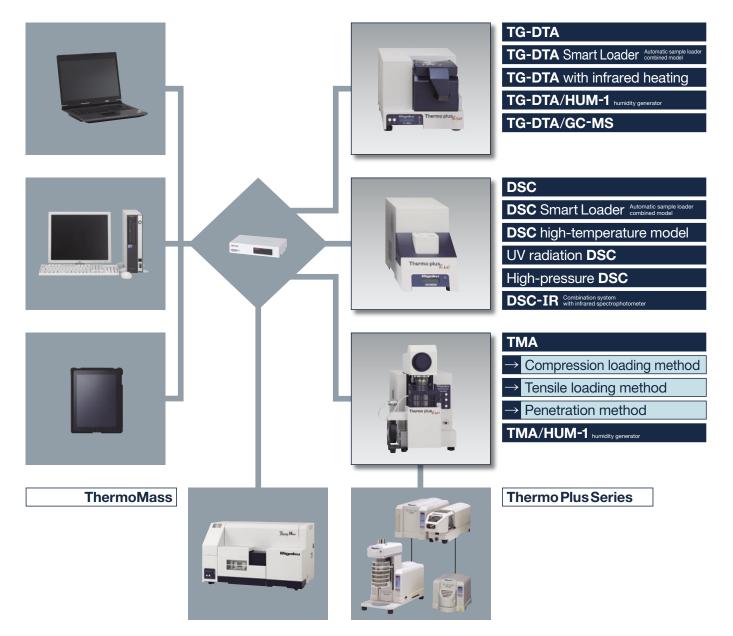


## Thermo plus EVOI Lineup and System Configurations

#### Thermo plus EVOI station

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The multi-task control and analysis software features a simple window layout, ease of use, and abundant display to meet a wide range of user needs.



#### Advanced measurement data protection

In the event of a problem resulting in a connection error between the station and module, the module will continue to perform measurements and store the measurement data after measurement.

The stored data can be retrieved using a PC, preventing loss of valuable measurement data in the event of connection interruptions.

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#### ECO mode

The system can switch to ECO mode when it enters standby mode after completing measurements, thereby reducing power consumption when the equipment is in standby mode. The TG-DTA's ECO mode allows rapid transition from standby to stable measurement conditions.

TG-DTA E00 Temp.	ECO FAN 22.4 °C Reset
TG DTA	0.0mq Detail

### Thermo plus EVOI Measurement and Analysis Software

#### Mail function

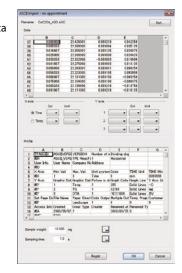
When connected to a company LAN, the Thermo plus EVO II software can send an email to indicate the completion of measurement or an error generation. It can also send measurement data to PCs, mobile phones, or other devices for immediate confirmation of measurement conditions at remote locations.

\* Requires connection to company LAN.

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#### Imports ASCII data

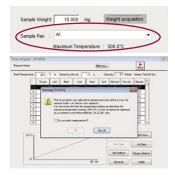
The software can convert existing measurement result data into ASCII format for analysis.



#### Sample pan alert

The new EVO series considers the thermal stability of any sample pan. Selecting a sample pan from the pull-down menu displays its maximum threshold temperature.

For example, if an aluminum pan is selected and more than 500°C temperature is set, a popup warning message will be displayed.



#### Automatic creation of module usage history file

The software automatically logs a history of module usage. It creates a file with date/time of usage, user name, model name, temperature program, and measurement result filename, among other data, for ready grasp of module usage and operating hours, contributing to equipment management and maintenance.

#### User-programmable end-of-measurement operation

Bata Nutr RAPI 160 RAPI 160 RAPI 160 RAPI 160

#### [End operation]

At the end of the measurement, the user can select end-of-operation settings such as ECO mode or Power OFF. Both settings are specified with temperature or time.

#### [Enforcement fan]

The enforcement fan settings enable you to activate the internal forced cooling fan: Used to the room temp ( cool-down to room temperature ), Not use ( inactivate ) or Normal use.

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	OK. Orest Antonio	

#### Exporting measurement data to Excel or Word

A menu command allows easy exports of data files to Word or Excel, while an associated layout edit function allows modification of export formats. When data is exported to Excel, numerical data are automatically entered in worksheet " Sheet 2," allowing data viewing and analysis with a wide range of other applications.



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#### First-aid function

If an error or a problem occurs in the module, the software displays an error No., error description, and first-aid procedure to facilitate rapid recovery of equipment operations. Descriptions of errors are also stored in a log file, which can be sent to Rigaku's Technical Service Department for prompt and accurate assistance.

	ce - [TG-DTA]		
Date :	2011/09/14	Module Status :	READY
]ime :	11:14:57	Nick Name :	TG-DTA
Type :	Error	Module :	TO-DTA
		Degice :	TG-DTA-TWN
		PAddress :	127.0.0.1
		Channel :	None
		Serial No :	8C30588A610F4
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# Thermo plus *Evo* II TG-DTA Thermogravimetric differential thermal analyzer



The combination of a highly regarded horizontal differential triple-coil balance system and a newly designed control and detection circuits results in accurate TG-DTA measurement results.

Compatible with a wide range of electric furnaces, this module is ideal for numerous types of measurement.

#### Horizontal differential TG-DTA

The horizontal differential triple-coil balance system boasts a proven track record for mass-change measurements. It corrects or cancels various types of fluctuations that can cause drifts. Combined with the newly developed electric circuits, it achieves low noise and excellent baseline stability.

#### Sample holder change

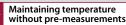
The plug-in sample holders are easily switched. Maintenance can be performed when the

sample holders need to be replaced due to sample boil-over or other reasons. Baseline adjustments can also be performed using the Adjustment window.

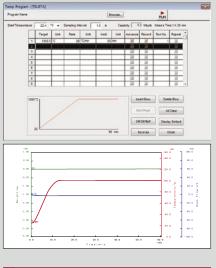
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#### Initial weight measurement

Using the zero-set function for tare reductions and the store function to save weight data in a file, users can use the module to measure initial sample weights.



Both conventional temperature and advanced temperature control modes are available. Temperature-hold control without overshooting is also possible without pre-measurements.

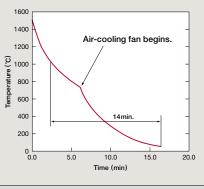


#### Mass calibration/temperature calibration

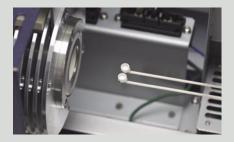
The calibration software supplied with the module allows TG mass calibrations using calibration weights, as well as temperature calibration based on the melting temperature of a standard calibration material or high-purity metal.

#### Post-measurement cooling

The air-cooling fan unit provided automatically begins operating after measurement to cool the module to room temperature. The fan cools the module from 1000°C to 50°C in 15 minutes for high throughput



Cooling time after measurements



#### Option

#### Analysis software

#### **TG Ozawa Method**

This method calculates activation energies based on three or more TG measurement results obtained at different heating rates. It then estimates the time required to reach a specific reaction rate.

#### Option

#### Infrared heating furnace

The infrared heating furnace can be selected to perform measurements at rapidly rising temperatures. It focuses infrared rays emitted from the infrared lamp to heat the sample. The low heat capacity of the electric furnace confers excellent temperaturehold characteristics after rapid heating, allowing measurement of changes before the target hold-temperature is reached. The system measures changes over time in

a temperaturehold condition. The water-cooling system for the furnace reduces cooling times after measurements.



#### Option

#### Attachments









#### Mass flow unit

The mass flow unit allows users to set the gas type and flow rate from the station. It can also be set to switch gases and stop gas flow in link with the measurement program.



Working with the measurement program, the gas selector switches the internal valves to control the gas to be flowed to the sample chamber. Flowmeters are optional. Please contact us for more information about flow rates and gas types.

# Gas flow unit

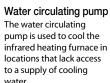
The gas flow unit supplies an inert gas to quickly lower the residual oxygen level in TG-DTA/TMA measurement. The unit can also supply air from its internal pump.

Flowmeter

The flowmeter controls the flow rate of the atmospheric gas (inert gas such as air) being supplied to the sample chamber. Three models are available (200/500/1000 mL/min).







Vibration-free stand

This vibration-free stand

suitable where numerous

external vibrations exist.

Wooden stand

of TG-DTA and TMA

Suitable for the installation

is designed for TG use,

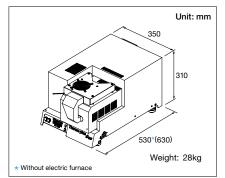
water.

#### **Specifications**

	Therm	o plus EVO I TG8120 series Therm	ogravimetric differential thermal a	nalyzer				
Model	TG-DTA standard model	TG-DTA standard model TG-DTA high-temperature model infrared heating *1						
Measurement temperate range	Ambient to 1100°C	Ambient to 1500 °C	Ambient to 950 °C	Ambient to 1500 °C				
Temperature rise rate	100°C/min Ambient to 700°C (120 sec.) *3 Ambient to 1000°C (60							
Measurement method	Horizontal differential triple-coil balance system							
Sample weight	Up to 1g							
TG range (FS)		500 mg						
TG resolution		0.1	μg					
DTA range (FS)		200	0 μV					
Measurement atmosphere		Air, inert gas flow, gas flow, re	educed pressure, water vapor					
Automatic sample loader *1		Samples : 24, Reference samp	les: 3, Calibration samples: 5					

\*1 Cooling water required. Tapwater, pressure of 0.2 to 0.3 MPa, 3 L/min. \*2 A 200-V power supply and the PU-4K power unit (option) are required. \*3 Stepwise heating

#### External dimensions



#### Station

Desktop PC, notebook PC, or tablet PC can be selected. An inkjet printer is provided.

#### Utilities

TG-DTA :

 $1\phi~100\,\text{VAC}\pm10\%~50/60\,\text{Hz},\,15\,\text{A}$  Grounding receptacle  $\times$  1

PC and peripherals :

 $4\,\text{A}$  (depending on the machine), Grounding receptacle  $\times\,4$ 

Standard infrared heating furnace\* :  $1\phi~100\,\text{VAC}\pm10\%~50/60\,\text{Hz},\,15\,\text{A}\,2\text{P}$  grounding receptacle  $\times\,1$ Tap water, water pressure of 0.2 to 0.3 MPa, 3 L/min

#### High-temperature infrared heating furnace :

1 $\phi$  200 VAC ±10% 50/60Hz, 20A Grounding required Tap water, water pressure of 0.2 to 0.3 MPa, 3L/min

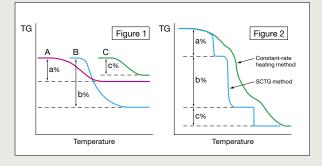
Can be used as a high-temperature furnace if the power supply is changed and the power supply unit (PU-4K) is added.

# Thermo plus <u>600</u> II Dynamic **TG**

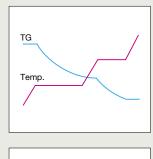
Dynamic TG is a sample-controlled thermogravimetry (SCTG) technique designed to perform temperature control with the rate of mass change as a parameter. With improved resolution for continuous TG changes, dynamic TG enables to ultimately monitor the mass change measurement with precision. Rigaku's Dynamic TG is equipped with 3 measurement modes as a standard, namely: constant reaction control (CRC) mode, stepwise isothermal analysis (SIA) mode, and dynamic rate control (DRC) mode.

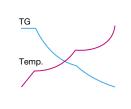
In conventional constant-rate heating TG, the time and temperature change simultaneously.

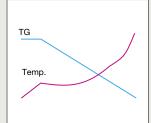
For example, 3 decomposition reactions, A, B and C simultaneously occurring in a parallel position, shown in Figure 1, the resolution and the quantitative separation have limitations. But with SCTG method, as illustrated in Figure 2, we can obtain a clear and distinct quantitative separation result.



#### Measurement modes







#### Stepwise isothermal analysis (SIA) First, the temperature is

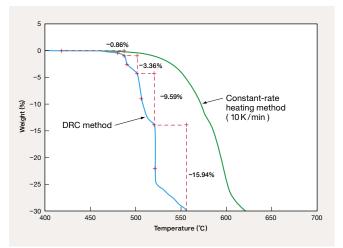
increased at a constant rate, and when the mass change rate (DTG) exceeds the preset value, isothermal control starts automatically. When the rate falls below the preset value, the temperature is increased at a constant rate. This sequence is repeated until the reaction ends.

#### Dynamic rate control (DRC)

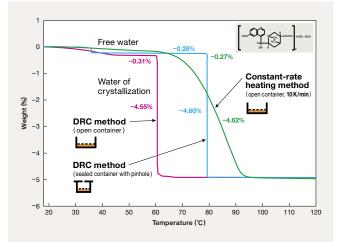
While the mass change rate is increasing, isothermal control is automatically performed. In response to a decrease in the mass change rate, the temperature rise rate is automatically increased. In comparison with SIA, DRC takes less time, so DRC is suitable for improvement of the quantitative separation. This control method is unique to Rigaku (patented).

#### Constant reaction control (CRC) To achieve a constant mass change rate, the temperature heating/cooling control is automatically applied. CRC is suitable for reaction mechanism examination, reaction-kinetic analysis, or the simulation of a reaction. CRC (patented) features the prediction of the measurement time.

#### Measurement example



Thermal decomposition of Vespel heat-resistant engineering plastic The graph compares the results of measurements of thermal decomposition of Vespel by the constant-rate heating method (10°C/min) and by dynamic TG (DRC mode). Results obtained by the constant-rate heating method do not clearly indicate phased decomposition; results obtained with dynamic TG indicate each phase of decomposition and yield accurate reduction rates. The evolved gas is then passed on to GC/MS for analysis. The constant-rate heating method detects a complex decomposition product resisting ready identification, while dynamic TG detects the main decomposition product for each phase: Teflon monomer, fluorosilane, benzene derivative, and aromatic compound (from the low temperature side to the high temperature side).



Separate determination of adhesion water, free water and crystal water in pharmaceuticals - Dehydration reaction of quinine sulfate dihydrate -Since moisture in a pharmaceutical significantly affects the stability, efficacy, and formulation properties, information on the bonding states of water and the amount of water in each bonding state is just as important as measurements of total amounts of water. The graph compares the results of measurement obtained by the constant-rate heating method and the DRC method, using an open pan and a sealed pan with a pinhole. The DRC method allows accurate control of sample temperatures, even at temperatures of 100°C or less, allowing separated determination of adhesion water, free water, and crystal water, even when the sample weighs several tens of milligrams—something previously difficult to perform. By performing measurements using an open pan and a sealed pan with a pinhole, we can simulate the thermal stability of dihydrates stored in open and sealed pans for assessments in compliance with the Japanese Pharmacopoeia.

# TG-DTA smart Loader Integrated TG-DTA Module with an automatic Intelligent Sample Loading system



This model consists of the TG-DTA module and a compact automatic sample changer that can load up to 24 measurement samples can be set in the sample changer. The reference sample can also be changed for each measurement. This system allows both continuous and single-sample measurements. Continuous measurements can be interrupted to perform other measurements. . The system can also reheat (2nd heating) the same sample.

#### TG-DTA sample changer

All Rigaku manufactured standard sample pans measuring 5 mm in diameter can be used. Three reference samples can be set to allow

DSC

selection of the appropriate reference sample based on measurement conditions. The cooling fan unit provided reduces cooling times after measurement for high throughput. Measurement conditions can be set using a single window for ease of use.



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2	18	Faish		~	46.8 10	teen 02_2	sample	10.000 mg	in Set	la Set	3	\$60.01C 20.01C/min
3	M.	Talah	. 1	-	40.0 10	test-03_2	sample	10.000 mg	in Set	la Set	3	\$60.0°C 20.0°C/min
	H			-	46.0 10	imat-04_4	sample	10.000 mg	in Get	In Set	4	560.0°C 20.0°C/min
5	R.	Wat	1	-	45.0 *0	test-05	Sanple .	10.000 mg	in Set	In Set	5	500.0°C 20.0°C/mm
5.7	16	Wat	1	1	42.0 12	Beal-06	sample	10.010 mg	in Set	la Sel	6	500.0°C 20.0°C/min
	R	Wat		-	45.0 10	test.07	sample	10.000 mg	in Set	In Set	6	660 0°C 20 0°C/min
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Program window

### Thermo plus & voll TG-DTA/HUM-1 Humidity Control TG-DTA



The compact humidity generator (HUM) connects to the TG-DTA for TG-DTA measurements in a water vapor atmosphere with constant relative humidity. This system is equipped with a polymer type relative humidity sensor and a high-precision temperature sensor for rapid response at varying vapor concentrations and for long-term stability. The system also allows temperature increase measurements under constant relative humidity.

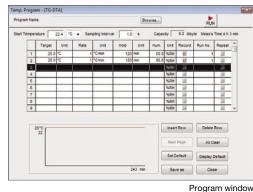
#### System configurations



When supplying water vapor at room temperature

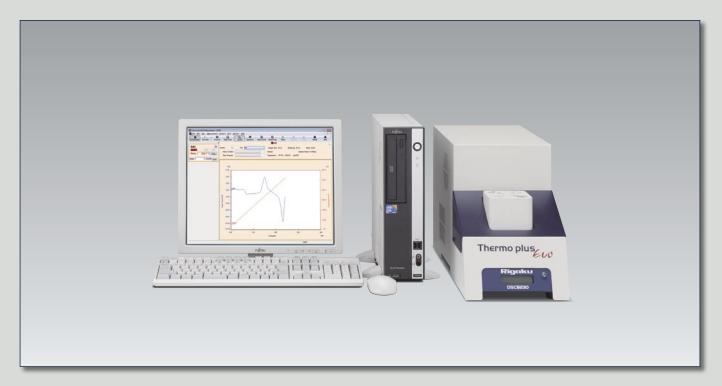
**Specifications** 

When supplying water vapor at temperatures above room temperature



Humidification method	Bubbling bath/dry gas combination method
Humidification range	Room temperature to 80 °C, up to 90%RH
Humidity sensor	Polymer type relative humidity sensor
Temperature measurement element	Pt resistive element
Humidification continuity	Approx. 100 continuous hours at 60 °C and 90%RH

Thermo plus *Evo* II DSC High-sensitive Differential Scanning Calorimeter

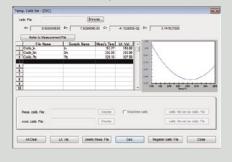


Featuring a new electric furnace and new electric furnace circuit technology, this system offers low noise and improved baseline stability.

This compact model requires an installation space as small as one and half sheets of A4-size paper.

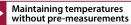
#### Temperature and Energy Calibration

A calibration table based on measurements of reference samples is used for temperature and energy calibration. The user can register several reference samples in the calibration table and different calibration files can be created. Temperature and energy calibration modes can be accomplished either by calibrating the temperature and energy during measurement or after the measurement is completed.



#### System expandability

This DSC system offers excellent expandability. It functions as a UV-DSC when an UV radiation attachment is mounted as a microspectroscopic DSC for sample observations or as an IR-DSC for simultaneous IR and DSC analysis when connected to an IR device.



Both conventional temperature and advanced temperature control modes are available. Temperature-hold control without overshooting is also possible without premeasurements.



Different DSC modules available for different needs

#### DSC8230HP

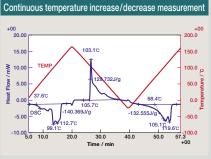
High-pressure DSC for measurements from ambient atmospheric pressure to 5 MPa

#### DSC8270

High-temperature DSC for measurements between ambient to 1500°C

#### Various cooling systems available

Users can select from a range of cooling systems based on the purpose of measurement and temperature range, including siphon, circulator, and liquid nitrogen auto feed types. The circulator or liquid nitrogen auto feed type cooling units allow continuous heating/ cooling measurements. Combined use with the DSC Smart Loader reduces cooling times after measurement.

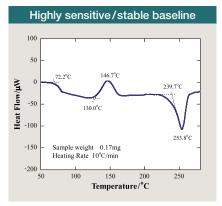


With liquid nitrogen auto feed type cooling unit

Melting and crystallization of polyethylene The graph shows measurement data for the melting and crystallization a branched linear lowdensity polyethylene (LLDPE). Since the melting of the sample during reheating depends on the crystallization that occurs during the cooling process, the pattern differs from the melting peak pattern in the first heating process.

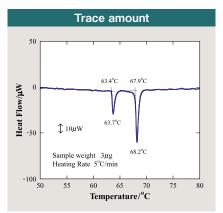
DSC

#### Measurement example



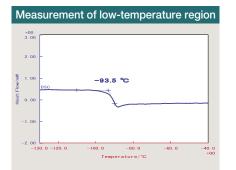
#### Glass transition, crystallization, and

melting of minute-amount amorphous PET The graph above shows the amorphous PET (sample amount: 0.17 mg) measurement results. The baseline is stable and the graph clearly shows the baseline shift ( $72^{\circ}C$ ), the exothermic peak ( $130^{\circ}C$ ) and the endothermic peak ( $239^{\circ}C$ ) due to glass transition, crystallization and melting, respectively.



#### Transition and melting of trace Dotriacontane The graph shows measurement results for dotriacontane (sample amount : $3\mu$ g), a linear-chain hydrocarbon. The graph clearly

shows an endothermic peak resulting from the transition on the low-temperature side and an endothermic peak resulting from melting on the high-temperature side.



#### Glass transition of butadiene rubber Generally, rubber is elastic at room temperature. But the butadiene rubber reveals a baseline shift due to glass transition near -93°C. At temperatures below the glass transition temperature, elastic materials such as rubber do not exhibit rubber characteristics instead they remain as amorphous solids.

#### Option

#### Analysis software

#### **DSC Ozawa Method**

This method calculates activation energies based on three or more TG measurement results obtained at different heating rates and estimates the time required to reach a specific reaction rate.

#### **DSC specific heat**

The specific heat capacity in each temperature is calculated based on the amount of baseline shift from 3 different measurement results namely sample pan, a sample with a known specific heat and an unknown measurement sample.

#### Purity

The melting peak of the sample and the melting peak of a high-purity substance are compared. The purity of a sample is calculated based on the melt fraction after correction.

#### Option

#### Various types of cooling units for a wide range of purposes



#### Siphon-type cooling unit

Liquid nitrogen is supplied to the cooling jacket through a siphon connected to a liquid nitrogen cylinder. This unit is applicable to temperature increasing measurements from low temperatures. Measurement temperature range: -150°C to 500°C



Liquid nitrogen auto feed type cooling unit The liquid nitrogen auto feed type unit is connected to the cooling unit and supplies liquid nitrogen continuously in response to a temperature program. It is suitable for heating and cooling measurements at a wide range. Measurement temperature range : -130 °C to 500 °C



#### Circulator-type cooling unit

A cooling system that continuously cools and circulates the cooling water of the circulator to the cooling unit. It can be used for continuous heating and cooling measurements. Tap water can also be used as cooling water.

Measurement temperature range : Temperature of the cooling water to 500 °C Heat circulating medium : Ethylene glycol (The cooling water temperature in F-25 by Julabo is -15 °C.)

#### Attachments



#### Sample crimper This sample pan crimper (packer) is used to make thermal contact between the sample and thermal

(specifically for sample containers ① and ⑯ for the DSC/TG-DTA)

plate.

#### Sample sealer

This sealer is used to seal a liquid, evaporative, or sublimative sample during measurement, specifically designed for sample pan (3). The sample sealer can withstand pressure of up to 0.3 MPa (3 atmospheres). (specifically for the DSC/TG-DTA)

High-pressure sample sealer The sealer on the left is specifically

for sample pan (1). The on the right

is for sample pan (1) ( conforming to

fire-prevention regulations). Both

sealers has a withstand pressure of

(specifically for use with the DSC/TG-DTA)

up to 5MPa (50 atmospheres).



#### **Specifications**







#### Flowmeter

The flowmeter controls the flow rate of the atmospheric gas (inert gas such as air) being supplied to the sample chamber. Three models are available (200/500/1000mL/min).

#### Gas selector

Working with the measurement program, the gas selector switches the internal valves to control the gas to be transferred to the sample chamber. Flowmeters are optional. Please contact us for more information about flow rates and gas types.

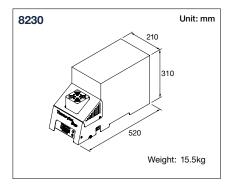
#### Mass flow unit

The mass flow unit allows users to set the gas type and flow rate from the station. It can also be set to switch gases and stop gas flow in link with the measurement program.

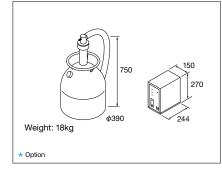
Model	Thermo plus EVO I differentia	I scanning calorimeter (DSC)
Model	8230	8270
Measurement method	Heat flux	method
Measurement temperature range *1	−150 °C ~ 725 °C ( up to 750 °C)	Ambient to 1500 °C
DSC range	$\pm 100 \mu\text{W} \sim \pm 100 \text{mW}$	$\pm 1 \mathrm{mW}$ $\sim$ $\pm 100 \mathrm{mW}$
Temperature rise rate (max.)	100 °C/min	20 °C / min
Noise level (RMS)	0.5 µW or lower	0.5 µW or lower
Measurement atmosphere	Air, inert ga	s, gas flow
Maximum sample amount	100 µL	50 µL
Cooling unit *2	Siphon type, circulator type, liquid nitrogen auto feed type	
Automatic sample changer *2	Samples: 24, Reference samples: 3, Calibration samples: 5	—
Pressure	Ordinary	pressure

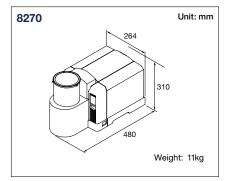
\*1 For temperatures below room temperature, an optional cooling unit suitable for the measurement temperature range is required. Inert gas flow is required for temperatures 500° or above. \*2 Option

#### **External dimensions**



Liquid nitrogen auto feed type cooling unit, control circuit \*





#### Station

Desktop PC, notebook PC, or tablet PC can be selected. An inkjet printer is provided.

#### Utilities

#### DSC :

1 $\phi\,$  100 VAC ±10% 50/60 Hz, 10 A Grounding receptacle  $\times$  1 PC and peripherals :

 $4\,\text{A}$  ( depending on the machine ), Grounding receptacle  $\times\,4$ 

Liquid nitrogen auto feed type cooling unit :

1φ 100 VAC ±10% 50/60 Hz, 3 A

# Thermo plus *Evo* II DSC smart Loader with Automatic Sample Loader



The DSC smart Loader consists of a DSC module and a compact automatic sample changer. A maximum of 24 measurement samples can be set in the sample changer. The reference sample can also be changed for each measurement. This system allows both continuous and single-sample measurements. Continuous programmed measurements can be interrupted to perform other measurements. The system can also reheat (2nd heating) the same sample.

#### DSC sample changer

All Rigaku manufactured standard sample pans measuring 5 mm in diameter can be used. Three reference samples can be set to allow

DSC

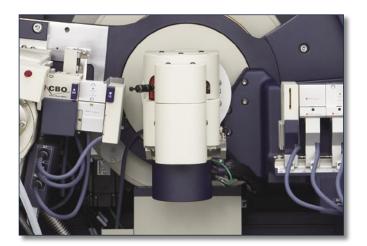
selection of the appropriate reference sample based on measurement conditions. Combined use with any of the available cooling systems allows configuration of a system suitable for a specific purpose. Measurement conditions can be set using a single window for ease of use.



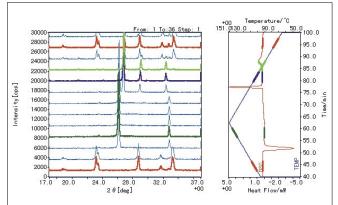
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1	K	Faist	3	15.0	Ŀ	40.	10	best-01	sample	10 030 mg is Set	IS Set	1	300 0°C 20 0°C mn
2	8		1	16.0	-	48.	10	Seat 02_1	eample	10.030 mg is Set	le Set	3	200.01C 20.01C min
3	M	Wat	1	15.0	-	40.	10	text-02	sample	10.000 mg to Set	Is Set	3	300 01C 20 01C/min
4	H	Wat		15.0	÷	40.	10	iest-04	eample	10.030 mg 16 Set	In Cel	4	365.0°C 35.0°C min
5	11	Wat		11.0		401	10	feat-05	sample	10.030 mg is Set	ta Set	5	300 01C 20.01Cmm
6	18	Wat	1	15.0	1	40.	12	leal-00	sample	10.030 mg in Sel	10.54	6	300 01C 20 01C/mit
7	R	Wat	. 1	15.0	Ŀ	42.1	10	Seat.07	aarpie	10.030 mg in Set	in Set	6	380.01C 20.01Cimin
8	18	Wat	· 1.	15.0	•	40.	10	Dest-08	sample	10.000 mg is Set	In Set	4	380-01C 20-01Cimin
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Program window

# Thermo plus *Evo* II X-ray DSC X-ray Diffractometer-Differental Scaning Calorimeter



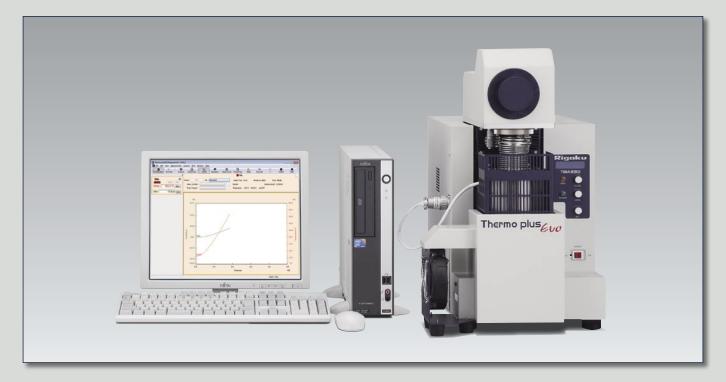
The X-ray DSC is a system where a DSC attached to a horizontal sample mounting X-ray diffractometer. This system can simultaneously measure DSC and XRD under the same temperature and in the same atmosphere. It can concurrently obtain information pertaining to temperature and energy, such as phase transition, and information on crystal conditions before and after the phase transition.



#### Phase transition of potassium nitrate (KNO3)

The potassion nitrate (KNO<sub>3</sub>) is a ferroelectric material used as a temperature standard sample in thermal analysis. Phase transition occurs from orthorhombic phase to rhombohedral phase during the heating process near 128 °C, then returns to the orthorhombic phase via an intermediate phase in the cooling process. The intermediate phase can be observed only during cooling. The graph shows the results of measurements of KNO3 during heating and cooling processes. The X-ray DSC system can easily measure thermal and structural changes in an unquenchable sample during the heating and cooling processes.

Thermo plus 600 II TMA Thermomechanical Analyzer



This TMA system adopts the differential expansion principle known for high-precision and accurate measurements. It can also be used for measurements by the compression loading, tensile loading, or penetration methods merely by changing the attachment. In addition, the system can perform measurements with the total single system to meet diverse measurement needs.

#### High-sensitivity and high-precision measurements using the differential system

This system adopts the Rigaku's established reputation on the differential expansion principle where the thermal expansion or shrinkage generated in the detection mechanism can be cancelled. The system offers high accuracy and excellent reproducibility in expansion and contraction measurements, even with low-expansion materials and thin samples. It can also perform measurements using the expansion system.

Supports a wide range of sample sizes

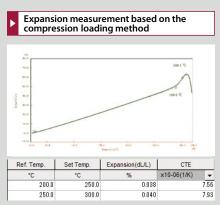
The differential TMA can handle samples of up to 9mm in diameter and 20mm in length.

Simple sample setting and automatic length measurement mechanism

Operating the switch enables to turn the loading on/off for changing samples easily. The sample length can be measured automatically and the user can record the length.

### Compact electric furnace for greatly improved heating/cooling speeds

The compact electric furnace provides excellent temperature response and allows temperatures to be raised at 100°C/min (up to 1000°C). After measurement, the cooling fan unit provided reduces temperatures from 1000°C to 50°C in 15 minutes.



Glass expansion, glass transition, hardening The graph shows the thermal expansion of a glass. The observed thermal expansion increased near 290 °C due to glass transition followed by hardening. Calculations of the expansion ratio and the coefficient of thermal expansion from 200 °C to 250 °C and from 250 °C to 300 °C show large differences in the expansion ratio and coefficient of thermal expansion before and after the glass transition.

#### Multi-measurement system with ultimate expandability

Multi-measurements such as expansion, compression, tensile loading and penetration, are made possible simply by replacing the attachment. For loading control, the analyzer supports constant loading, constant-rate loading (compressive/tensile loading is changed at a constant rate), and sine-wave cyclic loading ( compressive/tensile loading is applied with a constant frequency or oscillation ).

Calibration using sample with known expansion coefficient

Comparing the results of measurement of a sample with a known expansion coefficient and literature data allows users to produce and register a correction file containing the results of calculation performed using the difference in expansion rate at each temperature. Using this correction file to calibrate the expansion rates in the measurement results in more accurate measurement results, helping to manage the accuracy of data obtained with multiple systems.



DSC

#### **Product Lineup**

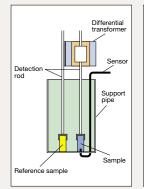


Auto cooling type TMA/L The TMA/L cooling unit continuously supplies liquid nitrogen as a coolant in response to a temperature program. The TMA/L can be installed as an attachment and it is best suitable for heating and cooling measurements between – 150°C to 600°C.

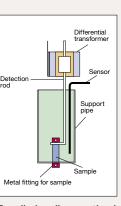


Horizontal TMA This TMA unit is a horizontal-type differential dilatometer. Measurement temperature range: Room temperature to 1000°C Sample size : 50 mm The support pipe and the detection rod are made of SiO2. Measurement range : ±2500 µm

#### Measurement methods



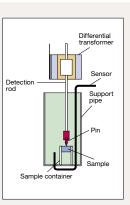
#### Compression loading method This method is used to measure the thermal expansion of a rod- or a plate-type sample. The differential expansion method is done by comparing the sample with a reference to eliminate any expansion of the sample support pipe. This method provides measurements with excellent reproducibility even for low-expansivity materials in the order of 10<sup>-7</sup>.



#### Tensile loading method This method is suitable for the measurement of fiber and film samples. Both ends of the samples are attached with a metal fitting and the tensile load is applied to the sample prior to measurement. This design cancels the weight of the metal fitting zipped on the sample, obtaining a high precision measurement.



Tensile loading method

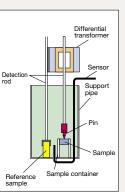


#### Penetration method

In this method, a pin is applied to the sheet or film material with a constant load during heating. It measures the temperature in which the pin penetrates into the sample and the degree of penetration. This method measures the temperature in which the pin penetrates into the sample and the degree of penetration. The penetration method can be applied during heating or isothermal depending on the objective of the measurement.



Penetration method

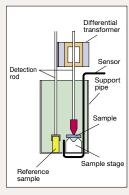


### High-sensitivity differential penetration method

The differential system unique to Rigaku is adopted in this method. This method enables high-precision measurement of the glass transition or softening of samples with a thickness in the order of micrometers. High sensitivity measurement is achieved ranging  $1\mu$ m to maximum scale.



High-sensitivity differential penetration method



#### High-sensitivity threepoint bending method

This method is used for bending-mode measurement of plate samples in which information on deformation temperatures as well as degrees of deformation are obtained. With the differential system employed in this method, it provides highsensitivity measurements.



High-sensitivity three-point bending method



ression loading method Compre (示差膨張)

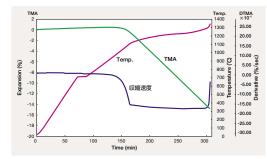
Compression loading method (全膨張)

d Tensile loa

12



Dynamic TMA is a temperature control method where the heating rate continuously changes, not by constant heating rate, but in response to the contraction rate in case of contraction associated with sintering of a sample. With this temperature control, the unit can be used to simulate temperature programs for obtaining sintered materials with inhibited grain growth.



Sintering of barium titanate at constant contraction rate

The sample was heated to 700 °C at 10 °C/ min, maintained at that temperature for 15 minutes, and then heated again at 5 °C/ min. This controls the contraction rate at 0.1%/min from the temperature at which the contraction rate was 0.5%.

#### **Specifications**

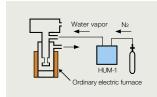
Measurement temperature range	Ambient to 1500 °C
Contraction rate control mode	<ol> <li>Constant temperature rise rate sintering mode</li> <li>Constant contraction rate sintering mode</li> <li>Quadratic function contraction control sintering mode</li> <li>Combination mode</li> </ol>
Contraction rate control range	0.02%/min $\sim$ 0.3%/min

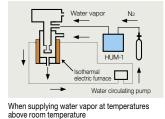
Other specifications specific to the TMA compression loading method / high-temperature type.

# Thermo plus Evol TMA/HUM-1 Humidity Controlled TMA



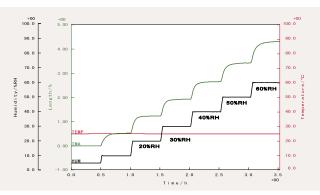
Connecting the compact humidity generator to the TMA module allows TMA measurements at a constant relative humidity condition. This system is designed to supply water vapor at room temperature or above. Users can configure systems to supply water vapor at room temperature or above. The module is equipped with a polymer type relative humidity sensor and a high-precision temperature sensor for long-term stability and for rapid response under varying water vapor concentrations. Relative humidity settings can be entered in the input window on the workstation. Control is synchronized with the temperature program. The system also allows temperature increase measurements under a constant relative humidity. System configurations





When supplying water vapor at room temperature

#### Measurement example



### Expansion of Nafion $^{\otimes}$ fluorine-based electrolyte membrane for fuel cells due to humidity changes

The graph shows the changes in expansion rates occurring when the temperature is maintained at 25 °C and the relative humidity (RH) is varied in stepwise from 5 % to 60 %. \* Nafion® is a registered trade mark of E.I. du Pont de Nemours and Company.

#### Specifications

Humidification method	Bubbling bath/dry gas combination method
Humidification range	Room temperature to 80 °C, up to 90 %RH
Humidity sensor	Polymer type relative humidity sensor
Temperature measurement element	Pt resistive element
Humidification continuity	Approx. 100 continuous hours at 60 °C and 90 %RH

DSC

#### Attachments



#### Flowmeter

The flowmeter controls the flow rate of the atmospheric gas ( inert gas such as air ) being supplied to the sample chamber. Three models are available ( 200/500/1000 mL/min ).



#### Mass flow unit The mass flow unit allows

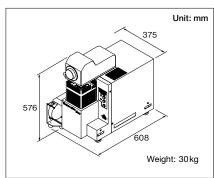
users to set the gas type and flow rate from the station. It can also be set to switch gases and stop gas flow in link with the measurement program.

#### **Specifications**

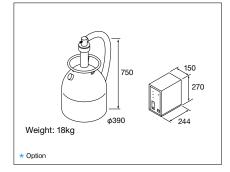
Model	Thermo plus EVOI TMA8310 series Thermomechanical Analyzer					
Measurement method	Compression loading method *1		Tensile loading method	Penetration method		
Туре	Standard mode	High-temperature model		Temperature-rising penetration	High-sensitivity differential penetration method	Isothermal penetration *2
Detection method used by the module	Differential expansion method		Single expansion method		Differential expansion method	Single expansion method
Standard sample size	$\phi$ 5mm (up to $\phi$ 9mm)		10 to 200 $\mu$ m in thickness	φ5mm		
	10 to 20 mm in length		0/15/20mm in length, 5mm in width	4 mm in thickness (maximum)		
Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>		SiO <sub>2</sub>	SiO2 (the pin is Ni)	
Maximum load	1000mN					
Measurement temperature range *3	Ambient to 1100°C	Ambient to 1500 °C	Ambient to 600℃			Ambient to 800 °C
Temperature rise rate (max.)	100°C/min ( low-temperature furnace : 20°C/min )					
Measurement range (FS)	5000 µm					
Loading mode	1. Constant loading ( up to 1000mN in the direction of contraction/tension, 1-mN steps ) 2. Constant-rate loading ( up to 1000mN at a rate of 1 to 500mN/min in the direction of contraction/tension ) 3. Sine-wave cyclic loading ( with an oscillation of 1 to 1000mN and a frequency of 0.01 to 1 Hz )					
Measurement atmosphere	Air, inert gas, or gas flow					

\*1 The specifications of the high-sensitivity three-point bending method conform to the compression loading method for the standard model. \*2 Viscosity measurement range: 10<sup>6</sup> Pa·s to 10<sup>10</sup> Pa·s (10<sup>7</sup> poise to 10<sup>11</sup> poise) \*3 Option : Low-temperature furnace (-150°C to 600°C), automatic liquid nitrogen supply attachment (-150°C to 600°C)

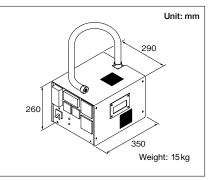
#### **External dimensions**



### Liquid nitrogen auto feed type cooling unit, control circuit \*



#### HUM



#### Station

Desktop PC, notebook PC, or tablet PC can be selected. An inkjet printer is provided.

#### Utilities

#### TMA :

 $1\phi~100~\text{VAC}~\pm10\%~50/60~\text{Hz},~15~\text{A}$  Grounding receptacle  $\times~1$ 

PC and peripherals : 4A ( depending on the machine ), Grounding receptacle × 4

Liquid nitrogen auto feed type cooling unit :

 $1\phi$  100 VAC ±10% 50/60 Hz, 3A

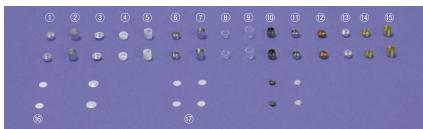
#### HUM :

1¢AC100V±10% 50/60Hz 5A

Varies with specifications/system configuration

# Sample pans for DSC/TG-DTA

To enable the flexible support of sample shapes, volume changes, and other measurement conditions, a range of sample pans is provided below.



- 1) Al  $\phi 5 \times 2.5$  mm 2) Al  $\phi 5 \times 5$  mm 3) Al for liquid, 30  $\mu$ L approx. 4) Alumina  $\phi 5 \times 2.5$  mm 5) Alumina  $\phi 5 \times 5$  mm 6) Pt  $\phi 5 \times 2.5$  mm 7) Pt  $\phi 5 \times 5$  mm 9) Quartz  $\phi 5 \times 5$  mm 9) Quartz  $\phi 5 \times 5$  mm 9) SUS (conformation with fireprevention regulations)
- 1) SUS (pan) AI (cover) 12 Cu  $\phi$  5 × 2.5 mm 13 Ag  $\phi$  5 × 2.5 mm 14 Au  $\phi$  5 × 2.5 mm 15 Au  $\phi$  5 × 5.5 mm 16 AI cover 17 Pt cover
- (1) is supplied with the standard configuration.)

## **Related Products**



#### ThermoMass Photo

Simultaneous measurement system of Thermogravimetry-differential thermal analysis and photoionization mass spectrometry

- Based on a patented skimmer gas introduction mechanism with a double orifice structure, the system provides high detection sensitivity, even with highly reactive gases.
- Equipped with a new fragment-free photoionization method (patent pending), electron impact ionization or photoionization measurement mode can be selected.
- With an all-in-one style where all components from the exhaust system to the gas introduction interface are integrated, the system is designed to be safe which also considers maintenance.

### TPD type R Photo / type V

#### Temperature programmed desorption evolved gas analysis

- The TPD type R Photo and the TPD type V are the lineup products in this category.
- The TPD type R Photo detects evolved gases in a specific atmosphere. It features a patented skimmer gas introduction interface with a double orifice structure equipped and has both electron impact ionization and photoionization measurement mode.
- The TPD type V detects evolved gases under high vacuum conditions.
- Both lineups support various sample shapes such as powders, bulk samples and plate materials.

\* The numeric values of performance indicated in this brochure are based

- on the test results at Rigaku. Rigaku does not warrant that the identical values can always be obtained regardless of different operational environments.
- \* Other company names and product names are trademarks or registered trademarks of the respective companies.

Specifications are subject to change without notice.



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