

## **Operation Manual**

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EXSTAR7000 Series High Sensitivity Differential Scanning Calorimeter





SII NanoTechnology Inc.

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## Foreword

Thank you for selecting the X-DSC7000 Differential Scanning Calorimeter.

This system performs Differential Scanning Calorimeter (DSC) measurements of inorganic materials such as ceramic and metals as well as organic materials such as high polymers metal. This system is designed for highly accurate measurements using simple operations within a wide temperature range of  $-150^{\circ}$ C to  $725^{\circ}$ C.

This manual is designed to assist the user in fully utilizing the performance of this instrument and was created with the purpose of enabling even first time users the ability to safely use the system.

In order to use this system safely and to its maximum potential, this manual should be read before the equipment is used. Please keep this manual nearby for easy reference.

When using this system, the software and option operation manuals are also necessary and should be kept near the system for reference as needed.

List of Reference Operation Manuals Muse Start-up Guide Sample Sealer Operation Manual Full Automatic Gas Cooling Unit Operation Manual Mass Flow Control Unit Operation Manual Muse Software Help

Do not use this system for any purpose other than for differential scanning calorimeter measurements.

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## 1. Safety Guide

#### 1.1. Definition of Signal Words in this Manual

Signal words are used in this document to denote unsafe situations that may arise based on certain actions. Cautions are placed as labels on the machine and are also used within this document. A definition of the terminology is provided below.

#### 

Indicates an immenently hazardous situation that, if not avoided, will result in death or serious injury.

### 🗥 WARNING

Indicates a potentially hazardous situation that, if not avoided, could result in death or serious injury.

## 

Indicates a potentially hazardous situation that, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices.

#### 1.2. Definition of Warning Labels on the Instrument

There are no hazzards if the instrument is used properly. However, failure to follow the instructions carefully or improperly handling the instrument increases the chance of fire or electrical shock.

The following labels are affixed to the instrument and are there to inform all who use, operate, or service the instrument of its potential danger and urge the individual to exercise caution.

There are three levels of warning ranked according to the relative danger of the condition.



Danger and warning are related to physical dangers that can harm the body.

Cautions are typically used for damage related to objects as well as to the body.

#### 1.3. Position of Warning Labels on the Instrument



Front



Back

#### Safety Precautions

#### 

- -Electrical Shock-
- This instrument employs a three-pronged power plug that includes a ground terminal. This ground terminal is the only ground used by the instrument. Improper grounding may result in electrical shock.
- Do not place hands or other objects into the cabinet vents or spaces. Failure to follow may result in electric shock.
- Be sure to shut off all power supplies when attaching options or performing maintenance procedures. Failure to follow may result in electric shock or other injury.
- Repairs, other than those directly specified in the Operation Manual or on warning labels affixed to the machine or in instances specifically directed by SII NanoTechnology Inc representatives, should not be performed. Opening and removing covers other than those specifically directed by result in extreme danger and may result in electric shock or fire due to large amounts of voltage.

### 🗥 WARNING

- -Temperature-
- The furnace and furnace area can be either extremely hot or extremely cold immediately following measurement.
- In particular, the cover can be extremely hot when the temperature is held for extended periods of time at above 600°C. Even if the samle temperature drops below room temperature it can be very hot. Be sure to use gloves, etc and do not touch with bare hands.
- Never touch directly with your hands to prevent burns or cold burns.

## 

-Gas-

- Do not use the machine in combustible environments.
  Discharges such as those from relay circuits may result in fire.
- · Do not use explosive gas material in the machine.
- Set the purge gas pressure to 0.1MPa.
- Do not measure samples that produce harmful gases.

#### 🗥 CAUTION

-Cautions when Connecting the Auto Sampler-

- Do not place your face or hand near the auto sampler during operation.
- When moving or removing the auto sampler, perform initial transport position adjustment prior to use. If the machine is moved in a non-adjusted state, the system may be damaged.
- When connecting or removing the cable between the auto sampler and the module, be sure to turn off the power to both the auto sampler and the module.

#### **1.4. System Precautions**

The cautions listed below will assist you in using the instrument under the best possible conditions. Read each of the points below before operation.

- 1. Always use a flow of inert gas when raising the temperature above 600°C (Refer to 6.1 High Temperature Measurements.)
- 2. Do not measure samples that evolve decomposed cracked or corrosive gases. If you must measure these types of samples, follow the instructions listed in chapter six of this manual.
- 3. Do not subject the sample holder to excessive force.
- 4. Always use sample containers that match your measuring needs. Also, follow the temperature restrictions for your selected container. Whenever measuring, insert a temperature protect value that is less than the temperature restrictions related to your sample. (Refer to section 4.7.1 Protect Feature and 8.3 Selecting the Sample Container.)

## 2. Overview

### 2.1. Components

### 2.1.1. Standard Components

	Name	Pcs
Main Unit	X-DSC7000 Main Unit	1
Attachments	Power Cable	2m×1
	PC Station Communication	1
	Cable	
	Accessory Kit	1
	Operation Manual	1
	Packing List	1
	Inspection Sheet	1

#### \*Accessory Kit (52S-100) Details

Part No.	Part Name	Qty
SSC000E030	Open Type Sample Container (Al $\phi$ 5.2, h2.5)	50
SSC000E032	Open Type Sample Container Crimp Cover	50
560-107	Sample for Calibration (In)	1
580-137	Sample for Calibration (Sn)	1
56US-114	Standard Sapphire (	1
560-101	Tweezers	1
560-102	Spatula	1
50-625	Alumina for Reference	1

## 2.1.2. Options

Name	Name Components		Comments
Fully Automatic Gas Cooling	Cooling Control Card (Internal Equip)	1	
Unit	Cooling Cable	1	
	Insulation Material	1	
	Velcro Tape	1	
LN2 Vapor Gas Generator	Siphon for 30L	1	
System 30L	LN2 Tank 30L	1	
LN2 Vapor Gas Generator	Siphon for 50L	1	
System 50L	LN2 Tank 50L	1	
LN2 Vapor Gas Generator	Siphon for 100L	1	
System 100L	LN2 Tank 100L	1	
Electric Cooling Unit PS2	Electric Cooling Unit	1	
	Step-up Transformer	1	
	(100V/60Hz Regions) Sold Separate	1 Set	
	ON/OFF Box	1	
	Remote Cable	1	
	Needle Valve for dry gas	1	
	Teflon Tube	5m×1	
	3 Type Joint (Quick Connect)	1	
	Grease (for cooling head)	1	
	Insulation Material	1	
	Velcro Tape	1	

Fully Auto Cooling Unit	Electromagnetic Valve	1	(*When using jointly	
·	Mass Flow Card	1	with the gas control	
	Piping Materials	1 Set	unit. only 1 mass flow	
	Polyurethane Tube	5m×1	card is needed.)	
Mass Flow Control Unit	Mass Flow Card	1		
	Mass Flow Main Unit	2		
	Piping Materials	1 Set		
Flow Meter Unit	Flow Meter 150ml /min F.S.	1		
	Teflon Tube $\phi 6 \times \phi 4$	5m		
Gas Control Unit	Gas Control Unit Main Body	1	(*When using jointly	
	Electromagnetic Valve Dual System	1	with the auto cooling	
	(Inside the main unit)		unit. only 1 mass flow	
	Mass Flow Card	1	card is needed.)	
	Teflon Tube	5m		
	Pining Materials			
Auto Sampler	Main Unit	1		
	Trav	1		
	Tray Cover	1		
	Attachment Metal Fittings	2		
	Attachment Screws	8		
	Cover Adapter	1		
	Container for Auto Sampler	50		
	Crimp cover for Auto Sampler	50		
	Container Crimp Die Set for Auto	1 Set		
	Sampler			
	Power Cable	2m×1		
Photochemical Reaction	UV Irradiator	1		
Differential Calorimeter set	Light Guide	1		
PDC-7	Shutter Control Cable	$2m \times 1$		
	PDC-7 Jacket	1		
	Lid with Quartz	1		
	Heat Ray Cut Filter	1		
	Protective Glasses	1		
Sample Sealer Set	Sample Sealer Main Unit	1		
cample could out	Die Set	3 Types		
Electric Driven Sample	Main Unit	1		
Sealer Set	Die Set	3 Types		
	Power Cable	1		
Real View DSC	USB Camera	1	Cannot be used	
RV-1D	lens	1	simultaneously with	
	LED Ring Light	1	either the auto	
	Inner Lid for Observation	1	sampler	
	Outer Lid for Observation	1	photochemical	
	Camera Stand	1	reachtion calorimeter	
	USB Cable	$5m \times 1$	reachaidh daidhinidtal.	
	USB Cable	5m×1		

#### 2.1.3. Consumables

2.1.3.1. Sample Containers

	Material	Product Code	Outer Dimensions (mm)	Max. Capacity	Upper Limit Temp. (Pressure Upper Limit)
Open Type Sample	AI	SSC000E030	φ <b>5</b> .2	45µl	600°C
Container	Pt	50-024	h 2.5		1500°C
	Alumina	SSC515D001		25µl	1500°C
	Cu	50-036			220°C
	Al	SSC505D003	φ <b>5</b> .2	100µl	600°C
	Pt	50-035	h5.0		1500°C
	Alumina	SSC520B005			1500°C
Open Type Sample Container Crimp Cover	Al	SSC000E032			600°C
Open Type Sample	AI	SSC000E041			600°C
Container Open Cover	Pt	50-025	φ5.0		1500°C
Open Type Sample	Cu	50-037			220°C
Container Mesh Cover	SUS	50-038			220°C
Sealed Sample	Al	SSC000E031	φ6	15µl	500°C (3MPa)
Container	Ag	560-04	h4		600°C (5MPa)
	SUS	560-06			600°C (5MPa)
	SUS (Au plating)	SSC521D002			500°C (5MPa)
Simple Sealed Sample Container	AI	SSC000C008	φ <b>6.8</b>	7.5µl	500°C (0.3MPa)
	Alodine Coating Al	SSC000C009	h2.6		300°C (0.3MPa)
Container for Auto	AI	52-023P	ф <b>6.8</b>	15µl	600°C
Sampler			h2.5		
Cover for Auto Sampler	AI	52-023C			600°C

For items without a listed pressure limit, there is not one.

#### 2.2. DSC Measurement Principle

The DSC detector is designed for heat flux DSC measurements. The basic structure of the chamber is shown below.



Figure 2-1 DSC Detector Components

The heat sink, made of an excellent heat conductor accurately controls temperature by following the temperature program through the use of a heater. The heat flow from the heat sink is conducted from the heat-conducting surface through the thermal resistance material into the sample and reference holders and containers. This increases, or decreases, the temperature of the sample and reference.

The heat flow passing through the thermal resistance material is proportional to the temperature difference between the heat conducting surface on one end and the sample holder on the other end. The specially designed heat sink is accurately controlled to maintain a uniform temperature at the heat-conducting surface.

Therefore, the measured difference in heat flow through the sample holder and reference holder is proportional to the sample and reference temperature differences. In this way, a heat flux type DSC detects the temperature difference signal and outputs it as a DSC signal.