

# *ARES* *Rheometer*



## **Rheometrics Series User Manual**

Revision J  
Issued January 2006



© 1999 by Rheometric Scientific Inc.  
© 2003 – 2006 by TA Instruments—Waters LLC  
109 Lukens Drive  
New Castle, DE 19720

## Notice

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without notice.

TA Instruments may have patents, patent applications, trademarks, copyrights, or other intellectual property covering subject matter in this document. Except as expressly provided in written license agreement from TA Instrument, the furnishing of this document does not give you any license to these patents, trademarks, copyrights, or other intellectual property.

TA Instruments Operating Software, as well as Module, Data Analysis, and Utility Software and their associated manuals and online help, are proprietary and copyrighted by TA Instruments. Purchasers are granted a license to use these software programs on the module and controller with which they were purchased. These programs may not be duplicated by the purchaser without the prior written consent of TA Instruments. Each licensed program shall remain the exclusive property of TA Instruments, and no rights or licenses are granted to the purchaser other than as specified above.

# Important: TA Instruments Manual Supplement

Please click on the links below to access important information supplemental to this Getting Started Guide:

- [TA Instruments Trademarks](#)
- [TA Instruments Patents](#)
- [Other Trademarks](#)
- [TA Instruments End-User License Agreement](#)
- [TA Instruments Offices](#)

# System Configuration

This table should be filled out during system installation with assistance from the TA Instruments service/installation engineer. Please refer to the information below when contacting TA Instruments for customer support or service.

Parameter	Specification/Designation	Serial Number
Instrument	ARES	
Motor		
Transducer		
Accessories/Tools		
Environmental Control System	Oven	
	Fluid Bath	
	Peltier	
LN2 controller		
Chiller		
Circulator		
Other		

# Notes, Cautions, and Warnings

Throughout this manual, the following terms and symbols are used to draw attention to specific situations.

NOTE: A NOTE highlights important information about equipment or procedures.



**CAUTION:** A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

---



**WARNING:** A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

---

# Safety

## Do Not Attempt Service

Do not attempt to service this instrument, as it contains no user-serviceable components.

## *Required Equipment*

While operating this instrument, you must wear eye protection that either meets or exceeds ANSI Z87.1 standards. Additionally, wear protective clothing that has been approved for protection against the materials under test and the test temperatures.

## *Safety Notices*

The following notices are intended to draw your attention to situations that pose a risk to either your personal safety or the safety of the instrument. Although these notices appear at relevant points throughout this manual, they are repeated here for emphasis. Additionally, all safety notices that appear on the instrument are reproduced on page 9.



**CAUTION:** Read the operating and maintenance instructions that were supplied with your air dryer. Failure to properly operate and maintain your air dryer will result in extensive damage to this instrument.

---



**WARNING:** If this instrument is used in a manner not intended or specified in this manual, the protection provided by the instrument may be impaired.

---



**WARNING:** This is a high-torque motor. Turning on the motor while in dynamic mode causes the motor to snap to dynamic zero position at a high velocity. This can cause severe damage to the transducer and/or personal injury. To avoid damaging yourself and the transducer:

**Never turn on the motor while a sample is loaded.**

**Keep hands clear of the motor.**

---



**CAUTION:** Force Rebalance Transducers contain a precision air bearing that is equipped with a bearing lock, which prevents movement of the air bearing when no air is applied. To avoid damaging your transducer, familiarize yourself with the operation of the bearing lock (see the next topic), and observe the following cautions:

Do not apply power to the instrument when the bearing is locked.

Do not unlock the bearing unless air is applied to the transducer.

If the air supply must be intentionally interrupted, turn off instrument power and lock the bearing prior to removing air.

If the air supply is interrupted while the bearing is unlocked, do not touch the anvil until air is restored.

Maintain air flow to the transducer at all times to prevent contamination of the air bearing.

Failure to observe these cautions will result in damage to the transducer.

---



**WARNING:** Do not attempt to lift or carry the Test Station alone. Attempting to lift or carry the Test Station can result in serious personal injury or damage to the Test Station.

---



**CAUTION:** Never place any lower tool into the bath if the temperature of the lower tool is cooler than that of the bath.

Placing a tool into a warmer bath will result in expansion of the tool during use. After expansion, the tool may not be removable without damaging your bath.

We suggest that you partially insert the tool by placing a phenolic spacer between the upper lip of the lower tool, and the surface of the bath. Allow the lower tool temperature to match that of the bath, then remove the spacer and fully insert the lower tool.

---



**WARNING:** Fuses must be changed by a qualified electronic technician only.

---



**WARNING:** Prior to changing a fuse, ensure that AC power to the Test Station is OFF. Changing a fuse on a live electrical circuit can cause serious personal injury or death.

---



**WARNING:** For continued protection against fire hazard, replace only with a fuse of the same type and rating.

---



**WARNING:** The following procedure must be performed only by a qualified electronic technician.

---



**WARNING:** Ensure that AC power to the Test Station is OFF before attempting the following procedure (see step a). Changing a fuse on a live electrical circuit can cause serious personal injury or death.

---



**WARNING:** The Main Power Switch does not remove power to the oven or LN2 Controller. The main power cord (220V IN) must be disconnected from the Power Panel to completely remove AC power from the system.

---





**WARNING: HIGH VOLTAGE** is used in the operation of this instrument. **DEATH ON CONTACT** may result if operating personnel fail to observe safety precautions. Learn the areas of high voltage connections, and exercise care not to contact these areas when performing instrument calibration. Prior to working inside the instrument, remove all jewelry, turn off the power, and ground points of high voltage before touching them. Make adjustments using an insulated electronic adjustment tool. Do not make physical contact with any component inside the instrument while power is applied to the instrument.

---

## *Instrument Labels*

Label found on the Rear of Power Chassis

**WARNING:  
FOR CONTINUED PROTECTION  
AGAINST FIRE HAZARD,  
REPLACE ONLY WITH SAME  
TYPE AND RATING OF FUSE.**

Label found on the Front of Transducer (FRT only)

**INSTALL/REMOVE LOCK  
WITH AIR PRESSURE ON**

Label found on the Front of Motor Mount

**WARNING:  
KEEP HANDS AND LOOSE OBJECTS  
AWAY FROM MOTOR DURING  
TURN ON AND OPERATION.**

Label found on the Front of Oven

**DANGER  
HOT TOOLS AND SURFACES**

# Regulatory Compliance

## Safety Standards

For the European Economic Area: (In accordance with Council Directive 73/23/EEC of 19 February 1973, amended by Council Directive 93/68/EEC, on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1: 1993 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010: 1994 Particular requirements for laboratory equipment for the heating of materials + Amendments.

## Electromagnetic Compatibility Standards

For the European Economic Area: (In accordance with Council Directive 89/336/EEC of 3 May 1989, amended by Council Directive 93/68/EEC, on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

Immunity: EN 50082-1:1997 Electromagnetic compatibility - Generic immunity standard, Part 1: Residential, commercial and light industry.

Emissions: EN 55011:1998, Class A

## Technical Support

For technical support concerning TA Instruments Rheometric Series, please call:

**1-302-427-4070**  
**8 a.m. to 5 p.m. USA ET, Monday through Friday**

For technical support concerning Microsoft Windows, please call Microsoft Corp. at 1-206-635-7000 (USA) 6 a.m. to 6 p.m. USA PT, Monday through Friday. In other countries, call the relevant Microsoft international subsidiary office.

# Table of Contents

Important: TA Instruments Manual Supplement.....	3
System Configuration.....	4
Safety .....	6
Required Equipment .....	6
Safety Notices .....	6
Instrument Labels .....	9
Regulatory Compliance .....	10
Safety Standards.....	10
Electromagnetic Compatibility Standards.....	10
Technical Support .....	10
Table of Contents .....	11
<b>Chapter 1: Introduction.....</b>	<b>19</b>
Overview.....	19
Description of Instrument.....	19
Motors.....	19
Motor Modes .....	20
Transducers .....	20
Force Rebalance Transducer with Normal Force (FRTN1) .....	20
Standard Transducer (STD).....	20
Environmental Control Options .....	21
Principles of Operation .....	22
Control Computer.....	22
Motor .....	22
Transducer .....	22
Environmental Control System.....	23
Specifications .....	24
Motor Performance Specifications.....	26
Transducer Operating Specifications .....	27
<b>Chapter 2: Instrument Components: Identification and Operation.....</b>	<b>29</b>
Component Identification and Placement.....	29
Host Computer.....	31
Test Station.....	31
Test Station Front Assemblies and Controls .....	31
Motor .....	31
Motor On/Off Control .....	32
Motor/Oven Stop .....	32
Transducer .....	33

Stage .....	33
Manual Stage Control .....	33
Software Stage Control .....	34
Stage Rate Adjustment .....	35
Front Panel LCD Display .....	36
Actuator Bearing Locks .....	37
Standard Transducers (2K STD and 10K STD).....	37
FRT with Normal Force (FRTN).....	37
Procedure for Locking and Unlocking Air Bearings: FRTN1 and FRTN1E.....	39
NOTE: 2KFRTN1 Bearing Lock Orientation .....	39
FRT without Normal Force (100 FRT and 200 FRT) .....	41
Procedure for Locking and Unlocking Air Bearings: 100 FRT and 200 FRT .....	41
High Resolution (HR) and High Torque (HT) Motor .....	41
Low Shear (LS) Motor.....	42
Procedure for Locking and Unlocking Air Bearings: LS Motor.....	42
Test Station Rear Input Panel .....	43
Signal Panel.....	43
Signal Connections.....	43
Power Panel .....	46
Power Connections .....	47
Main Power Switch.....	47
Pneumatics Panel .....	49
Gas Inputs .....	49
Gas Pressure Specifications.....	49
Air Quality Specifications .....	50
Pneumatic Connections.....	50
Environmental Control Systems .....	53
Oven.....	54
Oven Temperature Control.....	54
Chiller .....	55
Connection .....	55
Software Configuration .....	55
Chiller Operation.....	55
Maintenance.....	55
LN2 Controller.....	57
Dewar Flask .....	57
Connection .....	57
Software Configuration .....	57
LN2 Controller Operation.....	57
Maintenance.....	58
Oven Gas Selection .....	58
Oven Operating Requirements .....	59
Oven Configuration in Orchestrator™ .....	60
Oven Operation .....	61
Oven System Status Indicators.....	62
Fluid Bath 2.....	63
Description.....	63

Installation of Fluid Bath.....	63
Circulator Connections .....	66
PRT Installation.....	66
Lower Tool Installation and Removal.....	67
Filling the Circulator.....	67
Fluid Bath 2 Configuration in Orchestrator <sup>TM</sup> .....	68
PID Loop Setup .....	69
Fluid Bath 2 Operating Requirements .....	70
Fluid Bath 2 Operation.....	70
Peltier.....	72
Description.....	72
Circulator Options .....	73
Selecting a Thermal Operating Range.....	73
Installation of Peltier.....	74
Circulator Connections .....	76
Filling the Circulator.....	77
Peltier Configuration in Orchestrator <sup>TM</sup> .....	78
PID Loop Setup .....	79
Peltier Operating Requirements .....	80
Peltier Operation.....	80
Humidity Cover .....	80
Fluid Bath.....	82
Description.....	82
Installation of Fluid Bath.....	82
Lower Tool Installation .....	84
Circulator Connections .....	85
Inlet Line Clamp.....	85
Filling the Circulator.....	85
Fluid Bath Configuration in Orchestrator <sup>TM</sup> .....	87
PID Loop Setup .....	88
Fluid Bath Operating Requirements .....	89
Fluid Bath Operation.....	89
<b>Chapter 3: Test Descriptions.....</b>	<b>91</b>
Introduction.....	91
Stress, Strain, and Viscoelasticity.....	91
Definition of Terms.....	91
Hooke's and Newton's Laws .....	91
Steady Shear Testing .....	92
Dynamic Mechanical Testing.....	92
Definition of Variables .....	92
Measurement Method .....	94
Data Correlation.....	95
Documents Describing Instrument Software Operation.....	97
Test Modes.....	98
Strain Controlled Dynamic Test Methods.....	100

Dynamic Single Point Measurement .....	100
Functional Description .....	100
Suggested Uses .....	100
Test Options .....	100
Dynamic Time Sweep .....	101
Functional Description .....	101
Suggested Use .....	101
Test Options .....	101
Frequency Sweep .....	102
Functional Description .....	102
Suggested Use .....	103
Test Options .....	103
Dynamic Temperature Step .....	104
Functional Description .....	104
Suggested Use .....	105
Test Options .....	105
Dynamic Strain Sweep .....	106
Functional Description .....	106
Suggested Uses .....	107
Test Options .....	107
Dynamic Temperature Ramp Test .....	108
Functional Description .....	108
Suggested Uses .....	109
Test Options .....	110
MultiWave Single Point; MultiWave Temperature Ramp .....	111
Functional Description .....	111
Test Options .....	113
Frequency/Temperature Sweep .....	114
Functional Description .....	114
Suggested Use .....	114
Test Options .....	114
Strain-Controlled Steady Test Methods .....	115
Steady Single Point .....	115
Functional Description .....	115
Direction .....	115
Suggested Uses .....	116
Options .....	116
Steady Rate Sweep .....	117
Functional Description .....	117
Data Collection Mode .....	117
Direction .....	118
Suggested Uses .....	118
Options .....	119
Strain-Controlled Transient Test Methods .....	120
Step Shear Rate .....	120
Functional Description .....	120
Suggested Uses .....	121
Test Options .....	121
Stress Relaxation (Transient Step Strain) .....	122
Functional Description .....	122

Suggested Uses.....	123
Test Options.....	123
Multiple Extension Mode .....	124
Functional Description.....	124
Test Options.....	124
Arbitrary Waveshape Test.....	126
Functional Description.....	126
Equation Syntax .....	127
Test Options.....	127
Thixotropic Loop (Rate Ramp).....	129
Description.....	129
Suggested Uses.....	129
Options .....	130
Torque/Normal Relaxation.....	131
Functional Description .....	131
Suggested Uses.....	131
Options .....	132
Force Gap Test.....	133
Description.....	133
Suggested Uses.....	133
Test Options.....	133
Steady Step Rate Temperature Ramp .....	134
Description.....	134
Suggested Uses.....	135
Options .....	135
Stress-Controlled Transient Test Methods .....	136
Constant Stress Test.....	136
Functional Description .....	136
Suggested Uses.....	137
Options .....	137
Stress Ramp Test .....	138
Description.....	138
Suggested Use .....	138
Options .....	138
Test Options.....	139
Steady PreShear.....	139
Delay Before Test .....	140
Manual Delay .....	140
Apply AutoTension .....	140
Automatically Start Test When On Temperature.....	140
AutoTension Adjustment .....	141
Analog Data Input .....	143
AutoStrain.....	144
Measurement Options.....	145
Correlation Delay.....	145
One Cycle Correlation.....	145
Strain Amplitude Control.....	145

**Chapter 4: Test Geometries and Formulas .....147**

Introduction ..... 147

General Test Tool Information ..... 147

- General Recommendations for Geometry Selection..... 147
  - Thin Films or Fibers ..... 147
  - Fluids, Suspensions and Emulsions..... 147
  - Solid Samples, Including Thermosets, Thermoplastics, and Elastomers ..... 147
  - Polymer Melts and Soft Solids..... 148
  - Thermosetting Resins and other Curing Studies ..... 148
- Testing Limits and Compliance ..... 148
  - Definition of Compliance and Stiffness..... 148
  - Determination of Operational Range ..... 149
- Possible Sources and Causes for Error ..... 150
  - Discrepancies in Sample Geometry ..... 150
  - Testing outside the Linear Region ..... 150
  - Temperature Variations..... 151
  - Other Factors..... 151

General Test Tool Installation ..... 152

- Upper Tool Installation ..... 152
- Lower Tool Installation: Motor Mount (Oven or Ambient) ..... 152
- Lower Tool Installation: Fluid Bath Mount ..... 152

Setting the Gap ..... 155

- Manual Zero..... 155
- Automatic Zeroing and Gap Setting..... 156
  - Zeroing the Gap..... 156
  - Setting the Gap ..... 156
- Comments Concerning the Gap Control Panel..... 156
  - Enabling the Gap Control Panel..... 156
  - Read Test Tool Gap Checkbox..... 157
  - Displaying the Instrument Gap..... 157
  - Max Allowed Force While Setting the Gap ..... 157

Test Tool Installation and Sample Loading ..... 158

- General Guidelines..... 158
- Parallel Plates..... 159
  - General Information..... 159
  - Operating Ranges ..... 159
  - Tool Installation..... 161
  - Sample Loading..... 161
- Cone and Plate..... 163
  - General Information..... 163
  - Operating Ranges ..... 164
  - Tool Installation..... 165
  - Gap and Cone Angle..... 165
  - Testing at Other than Ambient Temperatures ..... 166
  - Sample Loading..... 166
- Torsion Rectangular, New Design..... 167
  - General Information..... 167



Sample Dimensions .....	168
Clamping Torque .....	168
Operating Ranges.....	168
Coefficient of Thermal Expansion ( $\alpha$ ) .....	170
Tool Installation .....	170
Sample Loading.....	171
Torsion Rectangular, Original Design.....	175
General Information .....	175
Sample Dimensions .....	175
Operating Ranges.....	176
Coefficient of Thermal Expansion ( $\alpha$ ) .....	177
Tool Installation .....	178
Sample Loading.....	178
Couette .....	181
General Information .....	181
Operating Ranges.....	181
Tool Installation .....	183
Sample Loading.....	183
Vane Tool .....	184
Double Wall Couette .....	186
General Information .....	186
Operating Ranges.....	187
Tool Installation, Original Fluid Bath.....	188
Sample Loading, Original Fluid Bath.....	189
Tool Maintenance.....	189
Tool Installation, Fluid Bath 2 .....	192
Sample Loading, Fluid Bath 2 .....	192
Tool Maintenance, Fluid Bath 2 .....	193
Extensional Viscosity Fixture (EVF) .....	195
General Information .....	195
Description of the Tool.....	196
Sample Support.....	196
Preparing Samples .....	196
Installing the Fixture.....	198
Preparing the Instrument and Test.....	199
Loading Samples.....	201
Loading the Sample Using the Offset Technique .....	201
Running Experiments.....	201
Plotting Data.....	202
Helical Ribbon .....	204
General Information .....	204
Couette Analogy .....	205
Operating Ranges.....	206
Tool Installation .....	206
Sample Loading.....	206
Test Formulas .....	207
Dynamic Measurement Formulas .....	207
Steady and Transient Measurement Formulas .....	208

<b>Chapter 5: Calibration .....</b>	<b>211</b>
Introduction .....	211
Procedures .....	212
Calibration Intervals .....	212
Torque Calibration (for All Transducers) .....	214
Procedure .....	214
Normal Force Calibration (for All Transducers).....	219
Normal Force Calibration Procedure .....	219
Phase Angle Check.....	222
Principle.....	222
Procedure for 1K FRTN1, 2K FRTN1, 2K FRTN1E, 2K STD, and 10K STD Transducers .....	222
Procedure for 100 FRTN1, 200 FRTN1, 100 FRT, 200 FRT and 1K FRTN1.....	223
Strain Calibration Check .....	224
Procedure .....	224
System Check Using PDMS.....	226
Procedure .....	226
Temperature Calibration.....	228
Temperature Calibration using Orchestrator™ .....	228
<b>Chapter 6: Maintenance .....</b>	<b>231</b>
General Information .....	231
Routine Maintenance.....	231
Cable and Hose Inspection .....	231
AC Power Cords.....	231
Air Hoses .....	231
Air Dryer .....	232
Cleaning the Instrument.....	232
Lifting and Carrying the Instrument.....	233
Test Station.....	233
Special Maintenance .....	234
Service and Repair of the Test Station .....	234
Diagnostic LEDs .....	234
Troubleshooting Guide .....	236
Calibration.....	236
Operation.....	237
<b>Index.....</b>	<b>241</b>
<b>Appendix A: Complex Modulus Limits.....</b>	<b>251</b>
<b>Appendix B: Inertia and Compliance Fixtures .....</b>	<b>251</b>

---

---

# Chapter 1

## Introduction

### Overview

This manual describes the following instrument-specific features of the Advanced Rheometric Expansion System, ARES. The figure to the right shows the test station equipped with the Forced Convection Oven. For information concerning the operation of Orchestrator software, see the Orchestrator Online Help.

### Description of Instrument

The Advanced Rheometric Expansion System (ARES) is a mechanical spectrometer that is capable of subjecting a sample to either a dynamic (sinusoidal) or steady shear strain deformation, and then measuring the resultant torque expended by the sample in response to this shear strain. Shear strain is applied by the motor; torque is measured by the transducer. Strain amplitude and frequency are set by the operator, with the actual sample deformation determined by the measured motor, and transducer, displacement.



### Motors

There are 3 basic motors available for use in ARES. The High Resolution (HR) motor is a ball bearing, direct drive motor. It provides very precise and accurate motion in both dynamic and steady modes. The applied strain is essentially instantaneous.

The High Torque (HT) motor is similar to the HR Motor, but is designed to deliver much higher torque. The HT motor is intended for use in conjunction with the 10K STD transducer.

The Low Shear (LS) is most commonly used for fluids testing. The motor shaft is supported axially by a precision air bearing. This minimizes axial runout, providing the smoothest normal force data. It also has a special controller that allows extremely low shear rates to be applied during steady shear tests.

## *Motor Modes*

In **Dynamic Mode**, the motion is oscillatory. The motor begins all tests at the motor zero position, and drives symmetrically about motor zero (at the chosen frequency) to the commanded displacement (strain). The maximum angular deflection of the motor is 0.5 radians from either side of motor zero. When in dynamic mode, the instrument measures strain and torque.

In **Steady Mode**, the motor can begin a test from any position, rotating either clockwise or counterclockwise (as specified) at a specific rotational (shear) rate. When in steady mode, the instrument measures rotational rate, sample torque, and, if equipped with the appropriate transducer, normal force.

## *Transducers*

There are two types of transducers available for ARES. The Force Rebalance Transducer (FRT) is an active type transducer, and provides excellent resolution and temperature stability.

The Standard Transducer is a passive, spring type transducer that provides high frequency response with the ruggedness desired by QC labs. Several different ranges are available for each transducer type.

### *Force Rebalance Transducer with Normal Force (FRTN1)*

The Force Rebalance Transducer with normal force (FRTN1) consists of independent rotational (torque) and axial (normal force) servo control systems, each utilizing position feedback to maintain the FRT shaft (mass in contact with the sample) in a null position when no force is applied. When force is applied to the FRT shaft, the servo control systems drive the shaft back to null position. The electrical current required to drive back to null position is proportional to the amount of force applied. This current is converted to DC voltage, and scaled to become the force (torque) output of the transducer.

All Force Rebalance Transducers are dual-range. Selection of range is performed using Orchestrator software. Firmware versions 5.00.00 and higher automatically switch ranges during a test. FRT transducers are also available with normal force measurement capability. For the normal force option, the measurement range in tension (downward) is 60% that of compression (upward) due to the weight of the transducer shaft being supported by the normal force servo.

### *Standard Transducer (STD)*

The Standard transducer provides high frequency response with the ruggedness required by QC labs. They are generally used for solids and melts testing since they typically lack the low end sensitivity needed for fluids measurements. The Standard transducer utilizes a shaft that is supported by a torsion bar. Mounted to this is a moment arm. A position sensor on each end of the moment arm produces rotational position information. In response to rotation in a given direction, the output of one sensor increases while the other decreases. A torque signal is then derived by taking the differential between these two outputs. The torsion bar and moment arm are axially supported by a flat spring, allowing axial compliance. A third position sensor, mounted to the top of the spring assembly, is used to sense axial force.

## Environmental Control Options

This instrument can be configured to subject samples to various thermal environments. These options are, a Forced Convection oven, a re-circulating fluid bath, or a Peltier system. See Chapter 2 for details.

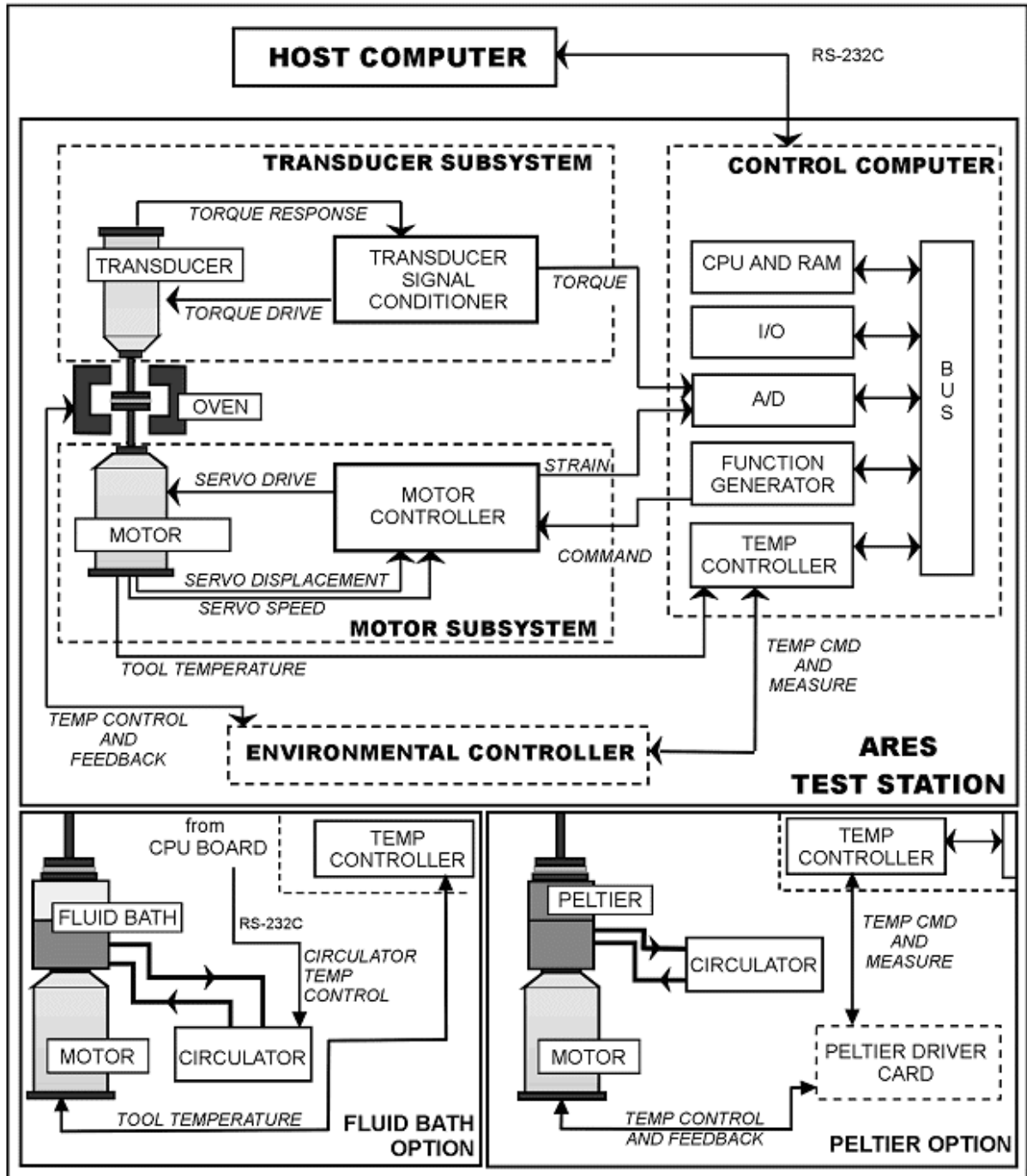


Figure 1-1. ARES: Functional Block Diagram

# Principles of Operation

Figure 1-1 is a simplified block diagram of the ARES. The following discussion offers a brief explanation of ARES operation, using Figure 1-1 for reference. In Figure 1-1, terminology printed in upper case refers to the various components or subsystems, and the signals generated by the system are printed in italics.

The ARES consists of the following subsystems:

- Control Computer
- Motor and Transducer
- Environmental Control

## Control Computer

The control computer synchronizes, generates and directs test instructions to, and processes raw data from, all subsystems. The control computer central processing unit (CPU) commands and monitors the control computer, maintaining communications via the bi-directional BUS. The input/output controller (I/O) controls all hardware switching within the instrument. The analog-to-digital converter (A/D) acquires analog signals from the motor and transducer, scales the signals for optimum gain, and converts them to digital form for processing by the CPU. The temperature controller (TEMP CONTROLLER) sends electronic commands to operate the environmental control subsystem (TEMP CMD AND MEASURE) based upon your input. It also acquires and processes temperature data (TEMP CONTROL AND FEEDBACK) for environmental control and display.

The CPU and random access memory (RAM) contain memory devices programmed to execute test sequences. The RAM circuitry stores data received from all subsystems for further processing by the HOST COMPUTER (IBM® or 100% IBM®-compatible PC), that communicates with the control computer through the RS-232C data communications link.

## Motor

In dynamic mode, the Motor is configured as a position servo. In steady mode it is configured as a rate servo. Following the start of a test, the FUNCTION GENERATOR determines the waveform to be applied (COMMAND). The MOTOR CONTROLLER uses the COMMAND signal (and the SERVO SPEED and SERVO DISPLACEMENT feedback signals) to drive the motor (SERVO DRIVE), applying deformation to the sample. A feedback displacement signal (SERVO DISPLACEMENT) is derived from a sensor on the motor shaft. SERVO DISPLACEMENT is conditioned and sent to the A/D input as the strain deformation signal (STRAIN).

## Transducer

The FRT Transducer is also configured as a position servo. The transducer shaft moves as a result of the sample deformation that is applied by the motor. A feedback displacement signal is derived from a sensor on the transducer shaft. The torque applied to the transducer is proportional to the energy required to hold the transducer shaft at a known position. This energy (TORQUE RESPONSE) is conditioned and sent to the A/D input as the force applied to the sample (FORCE).

The Standard transducer (not shown in Figure 1-1) generates a signal proportional to the movement of the internal torsion bar, without any active feedback control.

## *Environmental Control System*

This instrument can subject the sample to a number of thermal environments using several different environmental systems.

When using the forced air convection oven, two resistive heater guns, mounted on the left side of the oven, are used to control the sample test temperature. An optional liquid nitrogen controller allows testing at sub-ambient temperatures. The power to the heaters is directed and monitored by the ENVIRONMENTAL CONTROLLER, that receives its commands from the temperature controller (TEMP CONTROLLER) located within the control computer.

When a test temperature is selected at the HOST COMPUTER, the CPU configures the TEMP CONTROLLER to output a command (*TEMP CMD AND MEASURE*) to the ENVIRONMENTAL CONTROLLER. This command combines with the heater power feedback signals to increase or decrease heater power as necessary to control oven temperature. To monitor actual oven temperature Platinum Resistance Thermometers (PRT) are installed in the oven. Two PRTs monitor the air temperature by each heater and a third PRT (*TOOL TEMPERATURE*) monitors the temperature of the lower sample tool in use. The temperature controller electronics monitor PRT resistance to determine the actual internal oven temperature. A difference between the actual temperature as sensed by the PRTs, and the commanded temperature that is entered by you at the HOST COMPUTER, results in the generation of an error signal by the ENVIRONMENTAL CONTROLLER electronics (*TEMP CONTROL AND FEEDBACK*). This error signal either raises or lowers the internal oven temperature until the PRT-sensed temperature and the commanded temperature agree.

When using the Fluid Bath, the circulator is under RS-232 control from the Test Station. Either the fluid in the circulator (using the circulator's internal PRT), or the lower test tool itself (using the lower tool PRT) can be maintained at the desired temperature.

When the Peltier system is used, temperature control and monitoring is accomplished through the lower tool PRT connector (*TOOL TEMPERATURE*), and special electronics in the ARES Test Station.

# Specifications

Table 1-1. Physical Specifications

Chassis	Width	Depth	Height	Weight
Test Station	26 in	21 in	36 in	275 lbs
	66.0 cm	53.3 cm	91.4 cm	125 kg
LN <sub>2</sub> Controller (optional)	11 in	9.5 in	22 in	38 lbs
	28 cm	24.1 cm	55.9 cm	17.3 kg
Mechanical Chiller (optional)	29 in	20 in	46 in	260 lbs
	73.6 cm	50.8 cm	116.7 cm	118 kg
Fluid Bath Circulator (optional)	12 in	17 in	25 in	74 lb
	30.4 cm	43.1 cm	63.5 cm	33 kg

Table 1-2. Test Station Operating Specifications

Operating Parameter	Specification
Main Power	180 to 264 VAC @ 20 A , 50/60 Hz single phase
Transient Over-Voltages	Installation over-voltage category II
Temperature Range, Ambient	5° to 40° C
Relative Humidity	80% up to 35° C, then decreasing linearly to 50% at 40° C



Table 1-3. Pneumatic Ratings

ITEM	AIR INLET RATINGS		
	AIR PRESSURE	FLOW	AIR QUALITY
Instrument	80 psi (5.5 bar)	with Oven: 5.5 scfm (156 l/min)  without Oven: 3 scfm (85 l/min)	Instrument-quality air having the following characteristics: <ul style="list-style-type: none"> <li>• Any particles present in the air must be smaller than 5 microns in diameter.</li> <li>• Relative humidity = 35 to 70% Dewpoint = 10° C</li> </ul>
Air Dryer	100 psi (6.8 bar)	with Oven: 7.5 scfm (213 l/min)  without Oven: 5 scfm (142 l/min)	

Table 1-4. Accessories Power Specifications

COMPONENT	NOMINAL RATED VOLTAGE, CURRENT, AND FREQUENCY
Fluid Bath Circulator	USA: 115 VAC, 10 A, 60 Hz Europe: 220 VAC, 10 A, 50 Hz Japan: 100 VAC, 10 A, 50 Hz (60 Hz with boost transformer)
Mechanical Chiller	Standard: 220 VAC, 10 A, 60 Hz AC-PO Option: 200 VAC, 10 A, 50 Hz

Table 1-5. Operating Specifications, Environmental Controller

Operating Parameter	Forced Convection Oven	Fluids Bath	Peltier
Temperature Range Standard Optional	Ambient to +600° C -150° C to +600° C	-10° C to 150° C	-30° C to 150° C
Temperature ramp rate	0.1° to 50° C/min	Depends on Circulator used	30° C/min (at 20° C circulator fluid temp)
Temperature Stability at thermal equilibrium	± 0.5° C	± 0.01° C	± 0.1° C

## Motor Performance Specifications

Table 1-6. Specifications: High Resolution Motor (HR)

PARAMETER	DYNAMIC MODE	STEADY MODE
Angular Displacement Range	0.005 to 500 milliradians	Not Applicable
Frequency Range	1E-5 to 500 rad/sec	Not Applicable
Rotational Rate Range	Not Applicable	0.001 to 100 rad/sec

Table 1-7. Specifications: High Torque Motor (HT)

PARAMETER	DYNAMIC MODE	STEADY MODE
Angular Displacement Range	0.005 to 500 milliradians	Not Applicable
Frequency Range	1E-5 to 100 rad/sec	Not Applicable
Rotational Rate Range	Not Applicable	0.001 to 100 rad/sec

Table 1-8 Specifications: Low Shear Motor (LS)

PARAMETER	DYNAMIC MODE	STEADY MODE
Angular Displacement Range	0.005 to 500 milliradians	Not Applicable
Frequency Range	1E-5 to 100 rad/sec	Not Applicable
Rotational Rate Range	Not Applicable	1 X 10 <sup>-6</sup> to 200 rad/sec

## Transducer Operating Specifications

Table 1-9. Specifications: Force Rebalance Transducers 2K FRTN1 and 2K FRTN1E

TORQUE	SPECIFICATION
Measurement Range	High Range: 2 to 2000 g•cm Low Range: 0.02 to 200 g•cm
Maximum Operating Frequency	500 rad/sec version V8.x and above 100 rad/sec older than V8.x
NORMAL FORCE	SPECIFICATION
Measurement Range	2 to 2000 gmf

Table 1-10 Specifications: Force Rebalance Transducer 1K FRTN1

TORQUE	SPECIFICATION
Measurement Range	High Range: 1 to 1000 g•cm Low Range: 0.002 to 20 g•cm
Maximum Operating Frequency	200 rad/sec version V8.x and above 100 rad/sec older than V8.x
NORMAL FORCE	SPECIFICATION
Measurement Range	2 to 2000 gmf

Table 1-11. Specifications: Force Rebalance Transducers 100 FRTN1 and 200 FRTN1

TORQUE	SPECIFICATION 100 FRTN1	SPECIFICATION 200FRTN1
Measurement Range	High Range: 0.04 to 100 g•cm Low Range: 0.004 to 10 g•cm	High Range: 0.08 to 200 g•cm Low Range: 0.008 to 20 g•cm
Maximum Operating Frequency	100 rad/sec (16 Hz)	
NORMAL FORCE	SPECIFICATION 100 FRTN1 AND 200 FRTN1	
Measurement Range	0.1 to 100 gmf	

Table 1-12. Specifications: Force Rebalance Transducers 100 FRT and 200 FRT

<b>TORQUE</b>	<b>SPECIFICATION 100 FRT</b>	<b>SPECIFICATION 200 FRT</b>
Measurement Range	High Range: 0.02 to 100 g•cm Low Range: 0.002 to 10 g•cm	High Range: 0.04 to 200 g•cm Low Range: 0.004 to 20 g•cm
Maximum Operating Frequency	100 rad/sec (16 Hz)	
<b>NORMAL FORCE</b>	<b>SPECIFICATION 100 FRT AND 200 FRT</b>	
Not Used for Measurement		

Table 1-13. Specifications: Standard Transducers 2K STD and 10K STD

<b>TORQUE</b>	<b>SPECIFICATION 2K STD</b>	<b>SPECIFICATION 10K STD</b>
Measurement Range	0.2 to 2000 g•cm	1 to 10,000 g•cm
Maximum Operating Frequency	500 rad/sec (80 Hz)	
<b>NORMAL FORCE</b>	<b>SPECIFICATION 2K STD AND 10K STD</b>	
Measurement Range	2 to 1500 gmf	2-1500 gmf

---

---

# Chapter 2

## Instrument Components: Identification and Operation

### Component Identification and Placement

The base system (Figure 2-1) consists of the Test Station with oven, and Host Computer. Additional optional components include LN2 Controller and an air filter/dryer. Figure 2-1 shows the recommended arrangement.



**WARNING:** If this instrument is used in a manner not intended or specified in this manual, the protection provided by the instrument may be impaired.

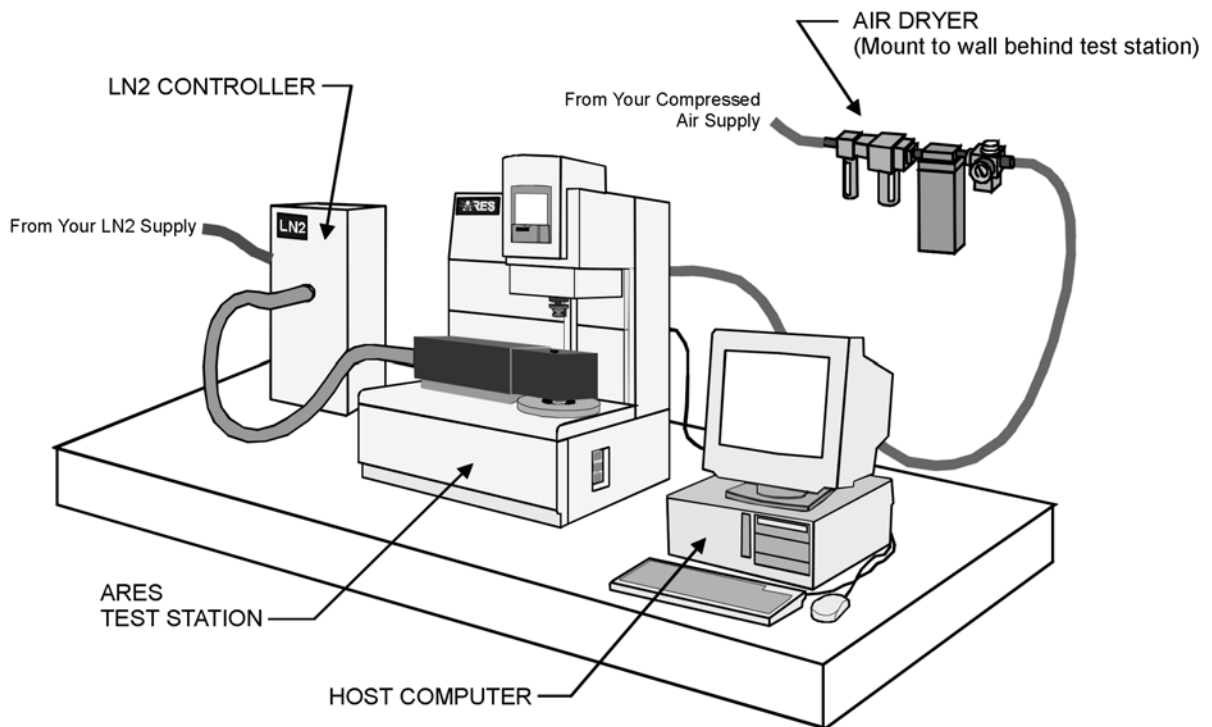


Figure 2-1. Base System

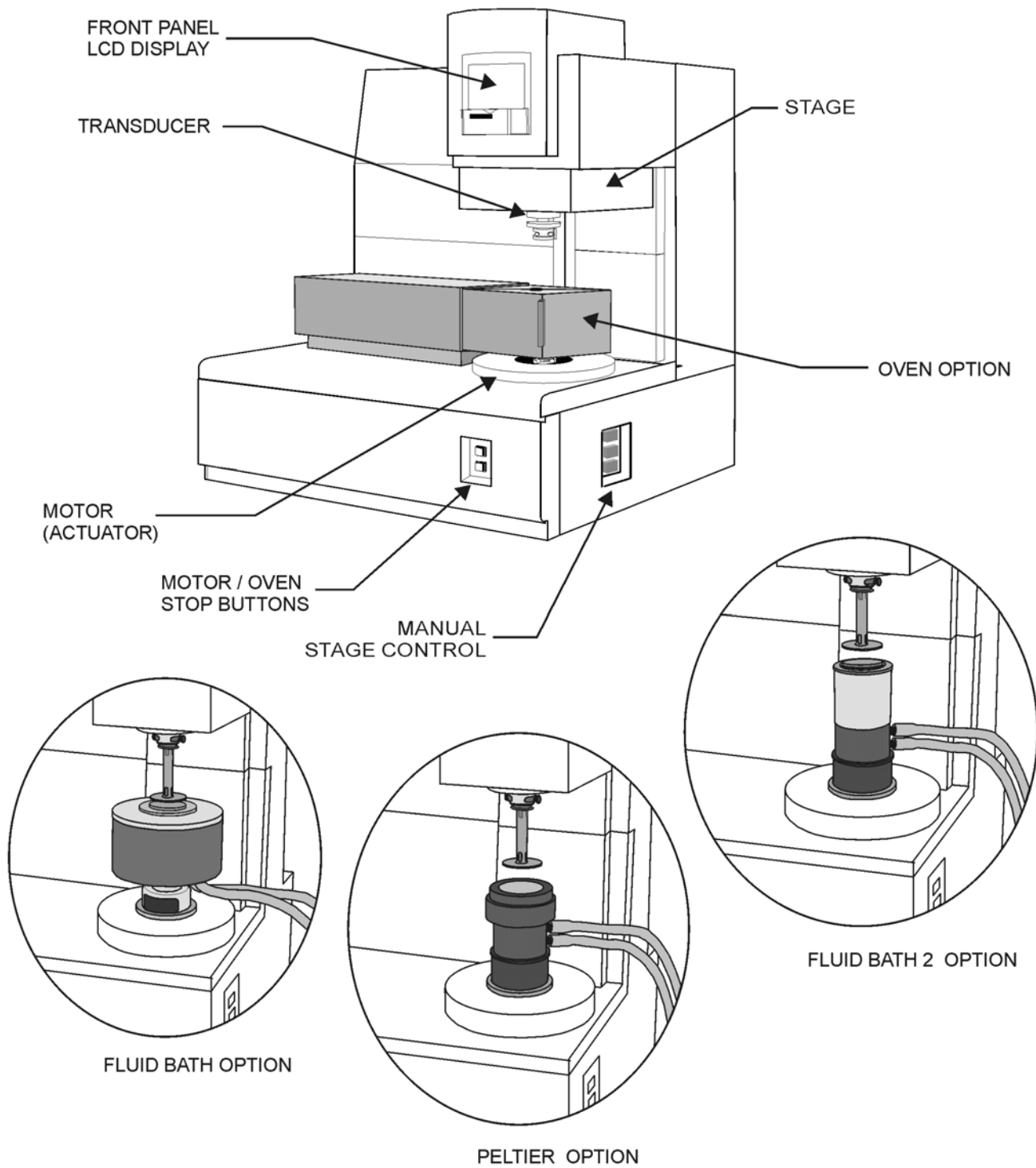


Figure 2-2. Test Station: Front View, Including Environmental Control System Options.

## Host Computer

The Host Computer allows the human operator to control the test station, and to monitor, display, and analyze data during the test. Control is achieved through Orchestrator software. While some basic references to the software will be made in the following sections, you should refer to the Orchestrator online help and other software references for a complete description of how to use the software.

## Test Station

The Test Station is the main instrument component that houses the motor and transducer, between which the sample to be tested is placed. It also houses the environmental controller, as well as the other electronic subsystems used in powering and controlling the Test Station. Figure 2-2 and Figure 2-11 show Test Station assemblies that are described in more detail within this chapter.

## Test Station Front Assemblies and Controls

The front of the test station (Figure 2-2) is where the motor, transducer and oven are located. In addition, the basic controls necessary to operate the test station are located on the front panel. All basic mechanical operations associated with the ARES are performed from the front of the test station. The following sections describe front panel systems in more detail.

## Motor

The Motor (Figure 2-2), also referred to as the actuator, deforms the sample by applying a shear strain to the sample. The Motor can be operated in either dynamic (sinusoidal) mode, or steady (constant rotational rate) mode. You can control the amplitude and frequency of the Motor movement.



**WARNING:** This is a high-torque motor. Turning on the motor while in dynamic mode causes the motor to snap to dynamic zero position at a high velocity. This can cause severe damage to the transducer and/or personal injury. To avoid damaging yourself and the transducer:

**Never turn on the motor while a sample is loaded.**

**Keep hands clear of the motor.**

---

## Motor On/Off Control

Power to the Motor is controlled using the *Instrument Control Panel* function available in Orchestrator software. To turn the Motor on or off, open the *Instrument Control Panel* by either clicking the "Control Panel" button (Figure 2-3), which appears on the Tool Bar, or from the Control pull down menu. Choose either MOTOR POWER ON or OFF, then select the "Ok" button.

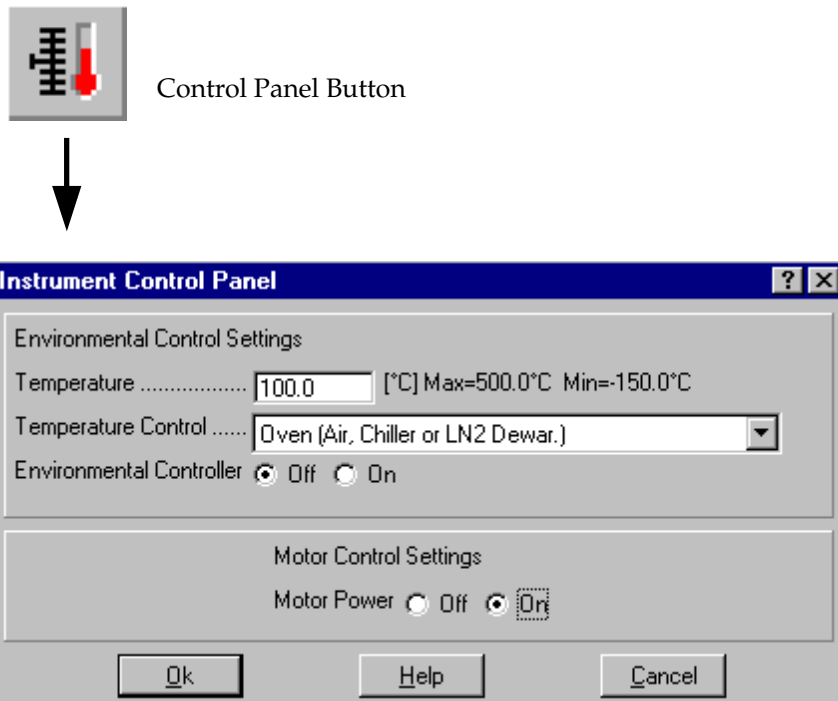


Figure 2-3. Instrument Control Panel

NOTE: If the Environmental Controller option is set "on", the Control Panel will appear as in Figure 2-20.

## Motor/Oven Stop

The Motor Stop and Oven Stop Buttons on the front panel of the Test Station will quickly and unconditionally power down their associated component. To turn the motor or oven back on, use the normal software controls.



# Transducer

The transducer (Figure 2-2) measures force generated by the sample during deformation by the motor. The sample is mounted between the motor and transducer using the various test tools described in Chapter 4.



---

**CAUTION: Force Rebalance Transducers contain a precision air bearing that is equipped with a bearing lock, which prevents movement of the air bearing when no air is applied. To avoid damaging your transducer, familiarize yourself with the operation of the bearing lock (see the next topic), and observe the following cautions:**

**Do not apply power to the instrument when the bearing is locked.**

**Do not unlock the bearing unless air is applied to the transducer.**

**If the air supply must be intentionally interrupted, turn off instrument power and lock the bearing prior to removing air.**

**If the air supply is interrupted while the bearing is unlocked, do not touch the anvil until air is restored.**

**Maintain air flow to the transducer at all times to prevent contamination of the air bearing.**

**Failure to observe these cautions will result in damage to the transducer.**

---

# Stage

The stage (Figure 2-2) is a motorized platform that supports the transducer. The stage can be raised and lowered to facilitate sample loading using either the manual stage control buttons on the right side of the test station, or through Orchestrator software control. The rate at which the stage moves can be adjusted through software.

## Manual Stage Control

The Manual Stage Control uses 3 push-button actuators located on the lower right side panel of the Test Station (Figure 2-2). It allows movement of the stage during operations such as sample loading. The movement of the stage is defined by the following modes of operation:

1. **Step:**  
When stepped, the stage moves very slowly, in single steps of the stage motor.  
To step the stage downward, press and hold the bottom button.  
To step the stage upward, press and hold the top button.
2. **Slew:**  
When slewed, the stage moves very quickly and smoothly.  
To slew the stage downward, press and hold both center and bottom buttons.  
To slew the stage upward, press and hold both center and top buttons.

## Software Stage Control

Movement of the stage can also be controlled from the *Set Gap/Instrument Control* function in the Orchestrator software. Click the "Set Gap" button (Figure 2-4) located on the tool bar. The *Set Gap/Instrument Control* function can also be opened from the Control pull down menu. The *Set Gap/Instrument Control* function has displays that show Torque, Normal Force and the current Gap. In addition there are several buttons that control instrument features. The "Send to Top" button can be used to move the stage all the way to the top of its motion. The "Set Gap" button can be used to move the stage to the entered commanded gap. The force used to move the tools together while setting the gap will not exceed the value entered in the "Max Allowed Force" box. The "Zero Fixture" button is used to bring the tools together to establish a zero gap reference, from which the gap is then set.



Set Gap/Instrument Control Button

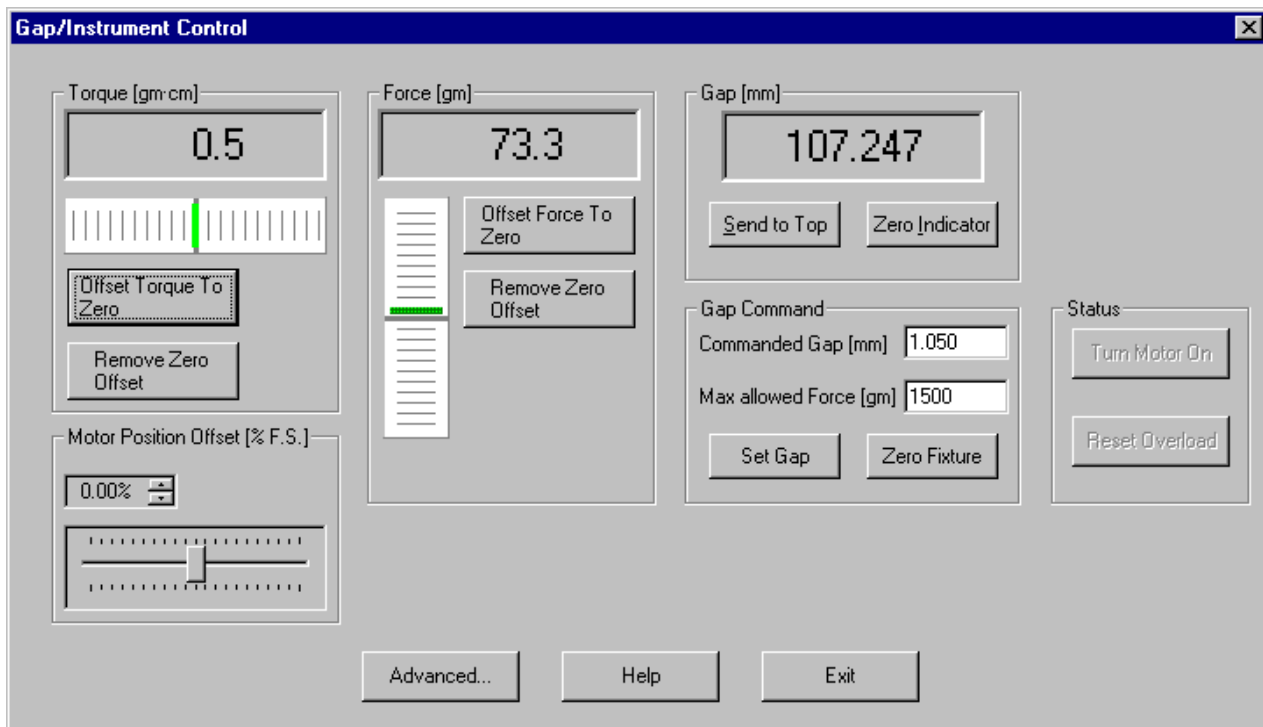


Figure 2-4. Set Gap/Instrument Control function main input form

## Stage Rate Adjustment

Both the operator-controlled step rate and slew rate of the stage can be adjusted through the *Advanced Control* form, shown in Figure 2-5. Click the "Advanced" button in the *Set Gap/Instrument Control* function main form to access the *Advanced Control* form. Adjust the step speed and slew speed as desired. Automatic stage movement rates, initiated using the "Send to Top" and "Set Gap" buttons, cannot be adjusted. The Front Panel LCD contrast is also adjusted from this dialog box.

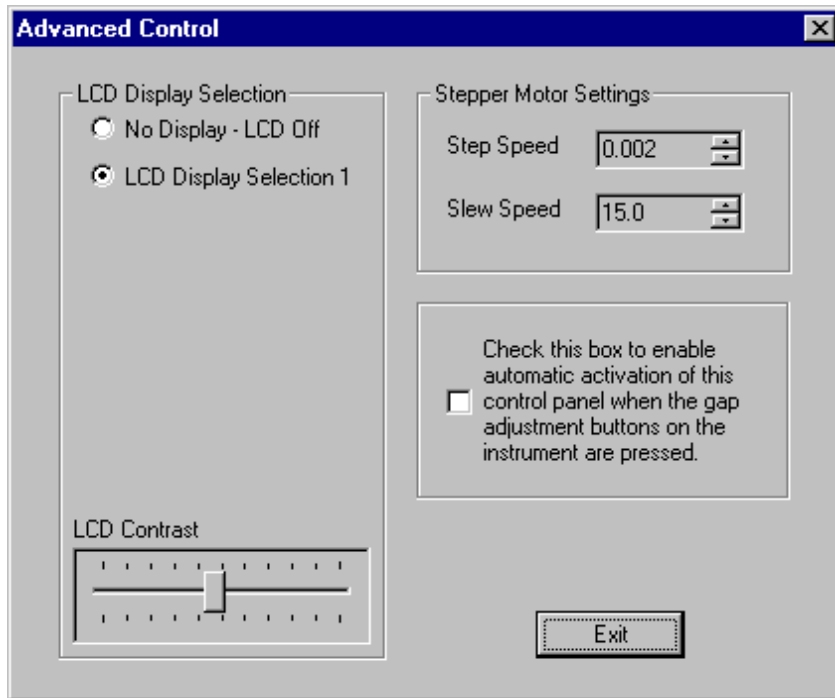


Figure 2-5. Advanced Control Form of the Gap/Instrument Control Function.

## Front Panel LCD Display

The front Panel LCD Display (Figure 2-6) is used to display certain instrument status information. For the ARES, temperature, gap, torque, and normal force are displayed. The LCD display can be turned on and off from the *Advanced Control* form of the *Set Gap/Instrument Control* function (Figure 2-5).

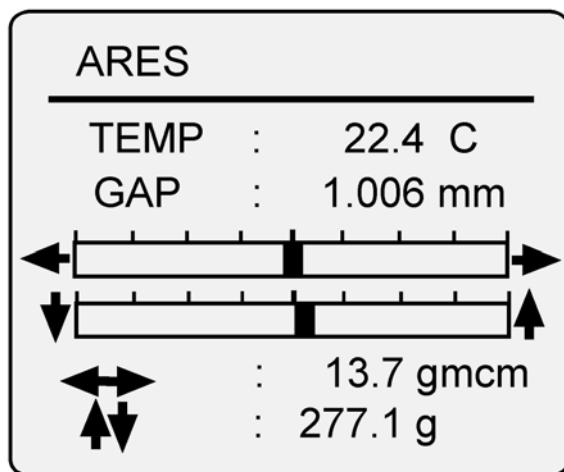


Figure 2-6. ARES Front Panel LCD Display

# Actuator Bearing Locks

## *Standard Transducers (2K STD and 10K STD)*

The standard transducers do not require a bearing lock.

## *FRT with Normal Force (FRTN)*

The FRTN transducers utilize a bearing lock consisting of a steel pin with a clamp on both ends (Figure 2-7) that is inserted through the transducer housing. On most 2K FRTN1 transducers, the pin is installed such that it points toward the left front corner of the instrument. However, some 2K FRTN1 transducers may be equipped with a pin that is rotated slightly from this position. The bearing locking and unlocking instructions for these transducers directs you to push the pin toward the left or right. Regardless of the orientation of your pin, “left” means toward the left hemisphere of the transducer, as viewed facing the front of the instrument. On low range transducers (100 FRTN1/ 200 FRTN1 and 1K FRTN1) the pin is installed so it points toward the front of the transducer housing, as shown in the lower half of Figure 2-7.

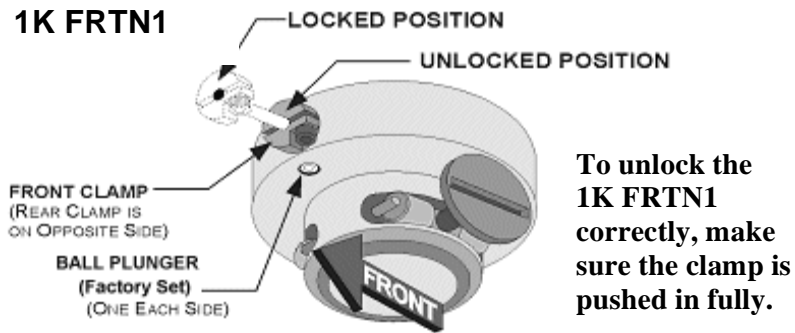
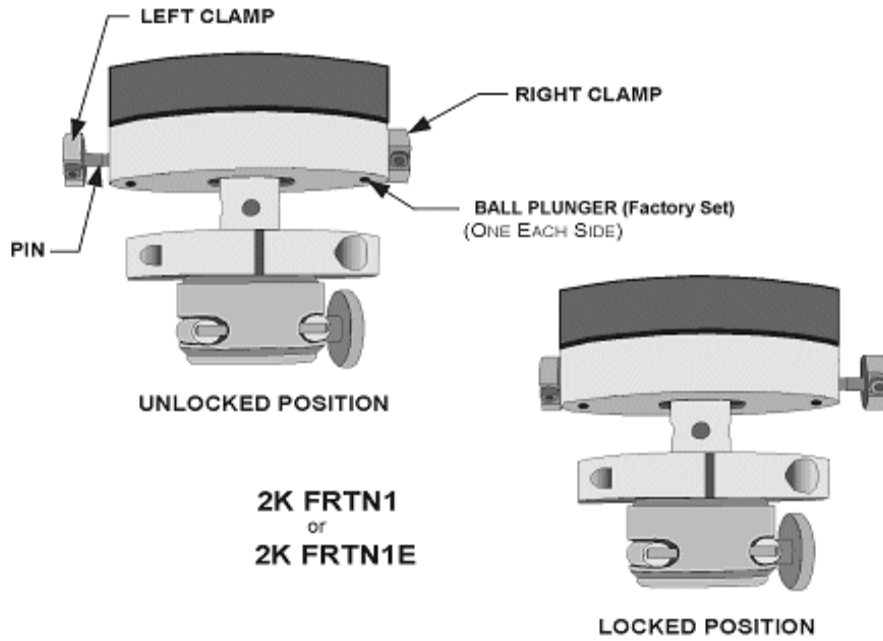


Figure 2-7. Bearing Locks: 2K FRTN1/FRTN1E and 1K FRTN1.

The view shown for the 2K FRTN1 transducers is looking from the front of the instrument. The lock on the 100/200 FRTN1 is similar to that shown for the 1K FRTN1 transducer.

## Procedure for Locking and Unlocking Air Bearings: FRTN1 and FRTN1E

Refer to Figure 2-7 while performing the following procedures.

To **Lock** the FRTN1 air bearing:

1. Read the Caution on page 7.
2. Ensure that instrument power is off and air is applied to the transducer.
3. Do one of the following depending on the transducer type:
  - **2K FRTN1 or 2K FRTN1E (delivered after January 2002; see note below):** Push the pin toward the right of the instrument until the left clamp contacts the transducer housing.
  - **100 FRTN1/200 FRTN1 or 1K FRTN1:** Push the pin toward the front of the instrument until the rear clamp contacts the transducer housing.

To **Unlock** the FRTN1 bearing:

1. Read the Caution on page 7.
2. Ensure that instrument power is off and air is applied to the transducer.
3. Do one of the following, depending on the transducer type:
  - **2K FRTN1 or 2K FRTN1E (delivered after January 2002; see note below):** Push the pin toward the left of the instrument until the right clamp contacts the transducer housing.
  - **100 FRTN1/ 200 FRTN1 or 1K FRTN1:** Push the pin toward the rear of the instrument until the front clamp contacts the transducer housing.

### NOTE: 2KFRTN1 Bearing Lock Orientation

During design revisions to the 2KFRTN1 transducer, the bearing lock direction was changed. Transducers delivered before January 2002 have the bearing lock arranged so that the locked position is with pin pushed to the left and the "right clamp" touching the transducer housing. Transducers delivered after January 2002 (approximately) are just the opposite; the locked position is with the pin pushed to the right and the "left clamp" touching the housing. It is very important to know which locking arrangement your transducer has.

In general, instruments delivered before January 2002 should have the older locking pin alignment. When locking and unlocking the bearings on these instruments, the directions described above should be reversed as shown in Figure 2-8.

If you are unsure as to which locking orientation your system has, the following procedure will help you determine it:

1. Turn off the power to instrument. Ensure that air is supplied to the transducer.
2. Holding the upper tool mount, manually try to move the transducer both axially (up and down) and rotationally.
3. Move the bearing lock to the opposite position and try to move the transducer again.

Note how each orientation feels as you try to move the transducer.

In the locked position, there will be no movement axially or rotationally. In the unlocked position, you should feel some movement in both directions. Make a note in your manual as to the locked and unlocked positions for future reference.

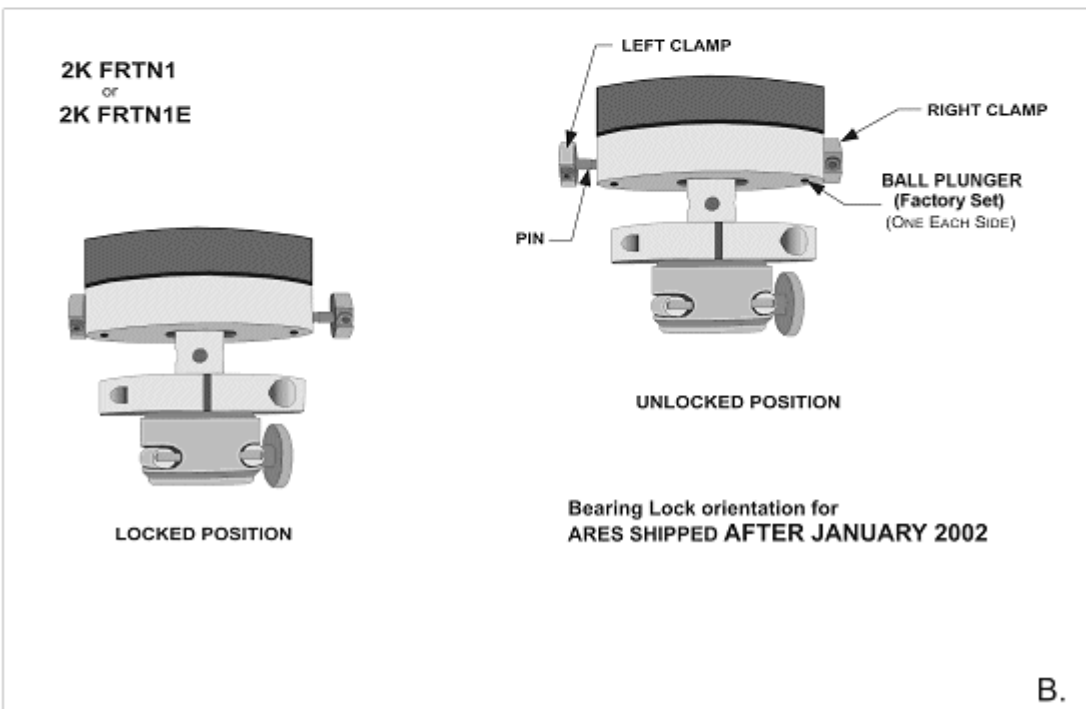
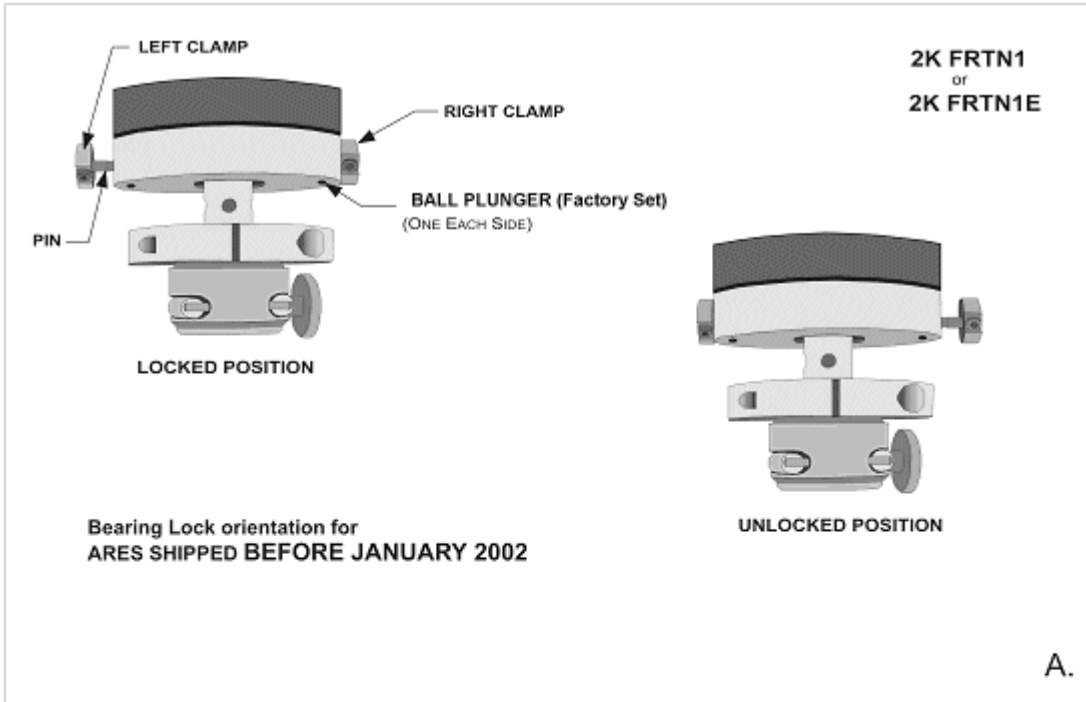


Figure 2-8. Bearing Lock Orientation: 2K FRTN1/FRTN1E.

The view shown is looking from the front of the instrument.

A. Systems delivered before January 2002.

B. Systems delivered after January 2002.



## *FRT without Normal Force (100 FRT and 200 FRT)*

The 100 and 200 FRTs use a bearing lock consisting of an aluminum block (Figure 2-9) that is fastened to the transducer by two machine screws. The block is machined at an angle that allows the bearing to be locked and unlocked by sliding the block.

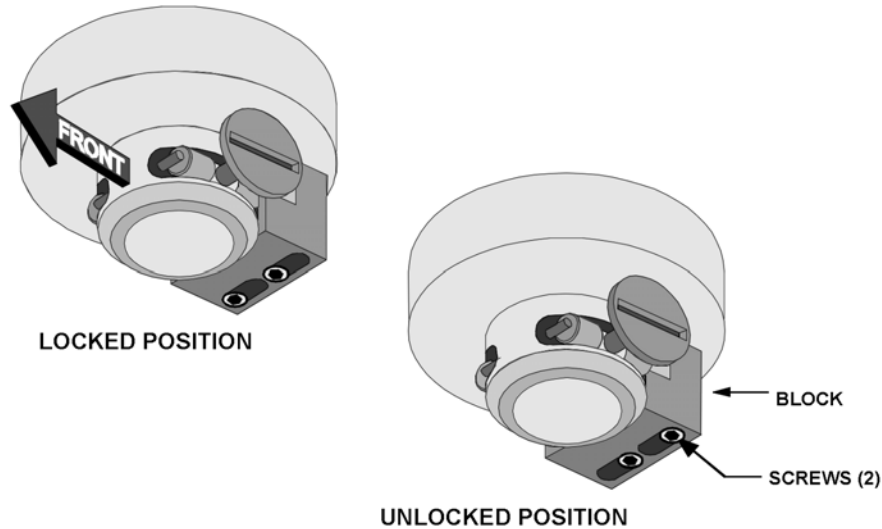


Figure 2-9. Bearing Lock: 100 FRT and 200 FRT

### Procedure for Locking and Unlocking Air Bearings: 100 FRT and 200 FRT

Refer to Figure 2-9 while performing the following procedures.

To **Lock** the FRT air bearing:

1. Read the Caution on page 7.
2. Ensure that instrument power is off and air is applied to the FRT.
3. Loosen both screws and slide the block to the locked position.
4. Tighten both screws.

To **Unlock** the FRT bearing:

1. Read the Caution on page 7.
2. Ensure that instrument power is off and air is applied to the FRT.
3. Loosen both screws and slide the block to the unlocked position.
4. Tighten both screws.

## *High Resolution (HR) and High Torque (HT) Motor*

The HT and HR motor do not require a bearing lock.

## Low Shear (LS) Motor

The LS motor bearing lock (Figure 2-10) consists of a formed metal plate that is fastened to the motor anvil with 3 machine screws and one Phillips head screw. To install this lock the motor cover must be removed.

### Procedure for Locking and Unlocking Air Bearings: LS Motor

Refer to Figure 2-10 while performing the following procedures.

To **Lock** the LS Motor air bearing:

1. Read the Caution on page 7.
2. Ensure that instrument power is off and air is applied to the Motor.
3. Remove the Motor cover (Figure 2-21, page 64), retaining 1 of the Phillips head screws for the lock.
4. Place the lock on the motor, aligning the 4 screw holes.
5. Inset the 3 machine screws and the Phillips head screw and tighten.
6. Retain the Motor cover and remaining Phillips head screws for re-installation when the lock is removed.

To **Unlock** the LS Motor bearing:

1. Read the Caution on page 7.
2. Ensure that instrument power is off and air is applied to the Motor.
3. Loosen and remove the 3 machine screws and the Phillips head screw.
4. Remove the lock.
5. Install the Motor Cover.

**NOTE:** the Phillips head screw removed from the lock is one of 4 screws used to secure the cover.

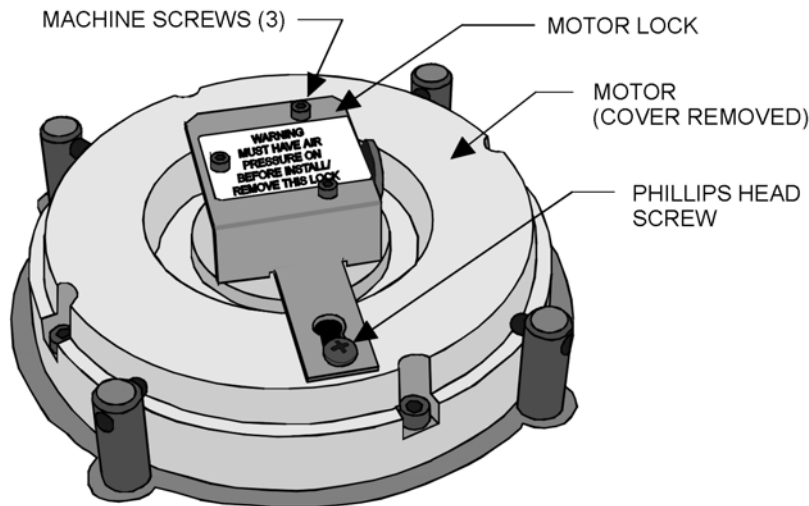


Figure 2-10. LS Motor Bearing Lock

# Test Station Rear Input Panel

The rear of the test station is where all electrical, pneumatic, and signal connections are made (Figure 2-11). The main power switch is also located at the rear of the test station.

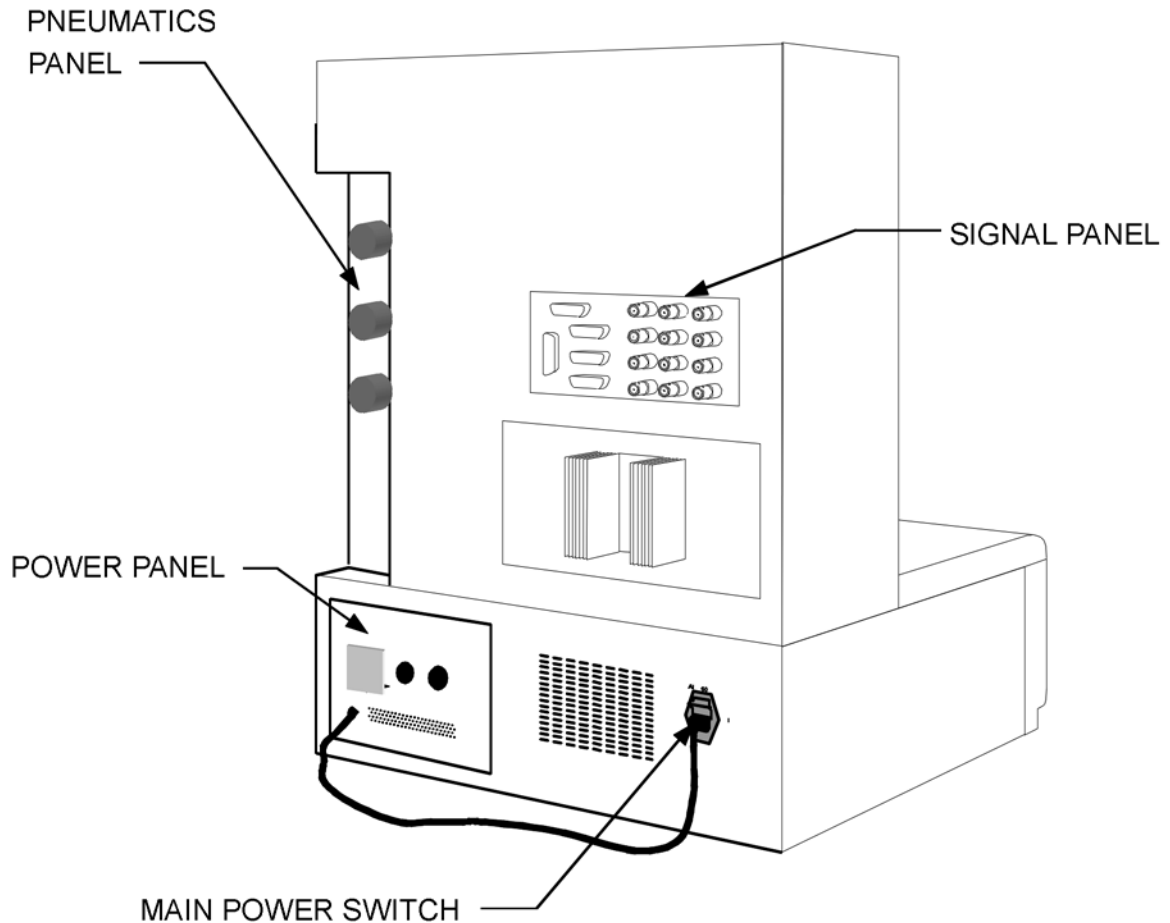


Figure 2-11. Test Station: Rear View

## Signal Panel

The Signal Panel is the input/output interface for electrical signals entering and leaving the Test Station. Figure 2-12 and Table 2-1 identify and describe Signal Panel connectors, including the basic connections for Test Station use.

### Signal Connections

There are two basic signal connections required for test station operation. They are a connection to the host computer, and a connection to the oven, or bath circulator, depending upon the environmental control system used. Connect the test station to the host computer through the HOST port on the signal panel, and the appropriate COM port on the host computer. By default, COM1 is selected by the software when it is run for

the first time. If you are using a different COM port, change the software configuration, using the *Instrument Set Up* function in the Utilities pull down menu, to reflect the correct COM port. You must tell the software the correct COM port you are using in order for communication with the Test Station to take place. If you have a serial mouse, more than likely it is using COM1, and you will need to use another serial port (COM2 for example) to communicate with the test station.

The oven position and temperature sensors are connected to the test station through the OVEN connector. The optional LN2 Controller is interfaced to the LN2 connector if cooling option is required. If the Fluid Bath is used connect the RS-232 output from the circulator to the CIRCULATOR connector.

Many of the connectors are not used under standard operation of the ARES. However, they are available for more customized applications when needed. Examples might be to plot applied strain on an external strip chart recorder, or to input an external voltage to be digitized with the test data. Table 2-1 contains more detailed information regarding these additional connectors.

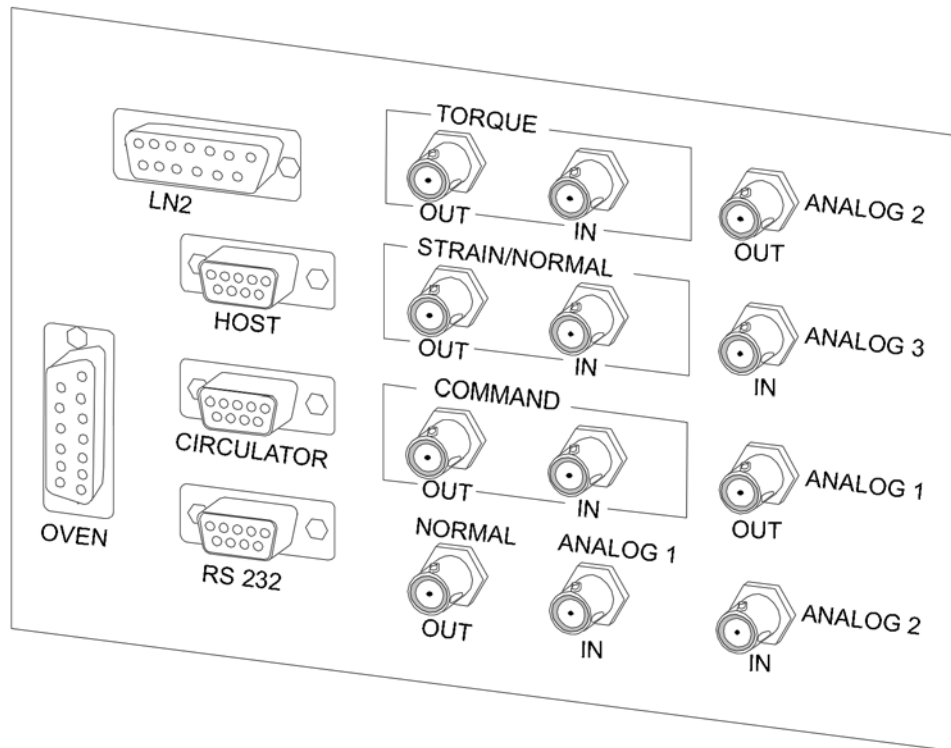


Figure 2-12. Test Station Signal Panel

Table 2-1. Signal Panel Connectors

Connector	Purpose
TORQUE OUT	Outputs a DC voltage that is proportional to transducer output (torque). Scaling is: 0 VDC $\approx$ 0 g-cm torque , $\pm$ 5 VDC $\approx$ $\pm$ full-scale torque
TORQUE IN	As the instrument is shipped, TORQUE IN is connected to, and outputs the same signal as, TORQUE OUT. This connector is normally used by TA service personel for diagnostic purposes.
STRAIN / NORMAL OUT	Dynamic Mode: Outputs a DC voltage that is proportional to motor strain (actual motor angular deflection). Scaling is: 0 VDC $\approx$ 0 radians, $\pm$ 5 VDC $\approx$ $\pm$ 0.5 radians Steady Mode: Outputs a DC voltage that is proportional to transducer normal (axial) force. Scaling is: 0 VDC $\approx$ 0 gmf , $\pm$ 5 VDC $\approx$ $\pm$ full-scale normal force
STRAIN / NORMAL IN	As the instrument is shipped, STRAIN/NORMAL IN is connected to, and outputs the same signal as, STRAIN/NORMAL OUT. This connector is normally used by TA service personel for diagnostic purposes.
COMMAND OUT	Outputs a DC voltage that is proportional to the selected strain, which drives the motor in dynamic mode. Scaling is: 0 VDC $\approx$ 0 radians $\pm$ 10 VDC $\approx$ $\pm$ 0.5 radians
COMMAND IN	As the instrument is shipped, COMMAND IN is connected to, and outputs the same signal as, COMMAND OUT. This connector is normally used by TA service personel for diagnostic purposes.
NORMAL OUT	Outputs a DC voltage that is proportional to transducer normal (axial) force. Scaling is: 0 VDC $\approx$ 0 gmf $\pm$ 5 VDC $\approx$ $\pm$ full-scale normal force
ANALOG 1 IN	Accepts a $\pm$ 10 VDC input signal that can be sampled at 1 Hz and stored in the data file. This is the input for the Analog Data input feature in Orchestrator
ANALOG 2 IN	For future use
ANALOG 3 IN	For future use
ANALOG 1 OUT	For future use
ANALOG 2 OUT	For future use
LN2	Communications interface between the optional LN2 controler and the instrument. Connected to LN2 controller.
HOST	Communications interface between the Host Computer and the instrument. Unless you have selected a different port using Orchestrator software, HOST is connected to the Host Computer serial communications port COM1.
CIRCULATOR	Communications interface between the optional fluid bath environmental control circulator and the instrument.
RS-232	Accepts a communication link that uses RS-232 protocol (used with OAM2 and DETA options).
OVEN	Electrical interface between the oven switches and temperature sensors and the instrument. Connected to the oven.

## Power Panel

For Test Stations equipped with an oven, the Power Panel serves three functions:

1. Electrical power interface for the Test Station.
2. Electrical power interface to the oven.
3. Electrical power interface to the optional LN2 Controller.

For Test Stations without an oven, the power panel is not required. Main power plugs directly into the Main Power Switch (shown in the next section).

Figure 2-13 identifies the Power Panel connectors.

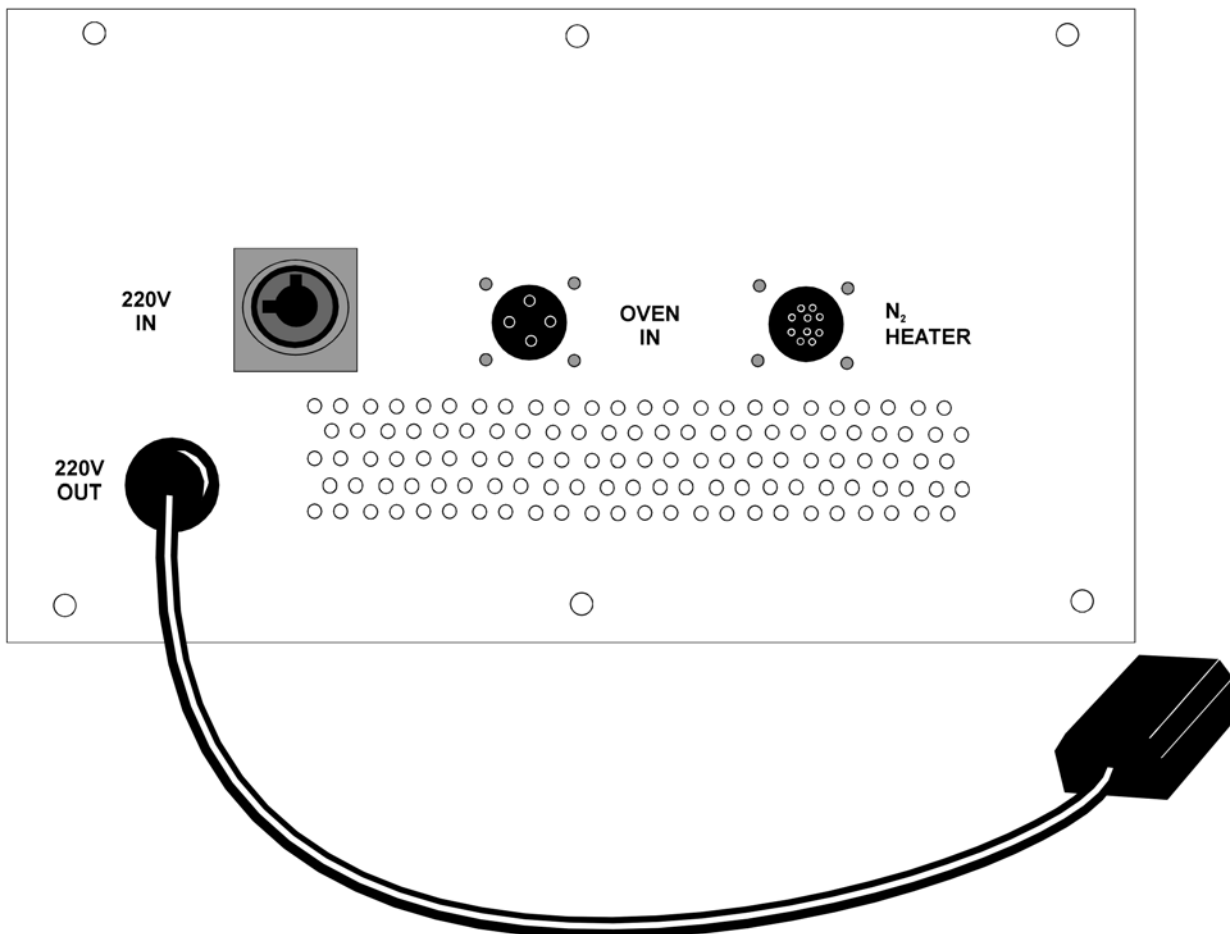


Figure 2-13. Power Panel.

## Power Connections

Power connections for the system are made as outlined in Table 2-2. Input power requirements are 220 VAC, 20A, 50/60 Hz.

Table 2-2. Power Panel Connectors

Connector	Destination	Purpose
220V IN	220 Volt input power supplied by customer	Main power to the system.
220V OUT	Connected to plug on Main Power Switch	Fused 220VAC power input to the test Station.
OVEN IN	Connected to Oven	220VAC power input to the oven control circuitry.
N2 HEATER	Connected to optional LN2 Controller	Power for the optional LN2 Controller.

### *Main Power Switch*

The Main Power Switch (Rear Panel) switches power on and off to the main portion of Test Station (Figure 2-14). The AC line fuse to the main portion of the test station is located below the power cord socket.



---

**WARNING:** The Main Power Switch does not remove power to the oven or LN2 Controller. The main power cord (220V IN) must be disconnected from the Power Panel to completely remove AC power from the system.

---

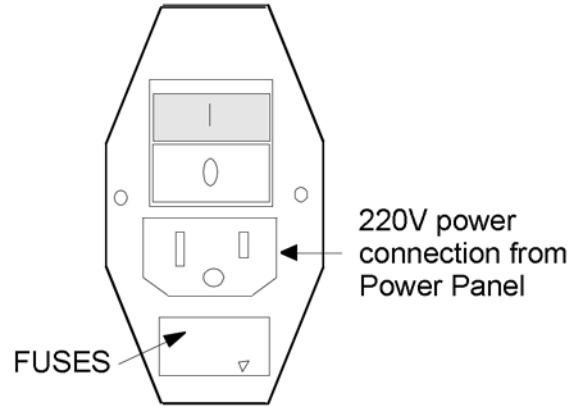


Figure 2-14. Main Power Switch



## Pneumatics Panel

The Pneumatics Panel is where all gas connections are made, adjusted, and monitored. This panel is located at the right rear corner of the Test Station. This panel is unique in that it is a two-sided panel, and wraps around the Test Station from side to back. Figure 2-15 shows both the side and back of the Pneumatics panel.

### Gas Inputs

Two gas inputs are available, one for standard compressed air (Air Supply -MAIN) and an optional port (N2/GAS) for other compressed gases. The main air supply serves two very important functions. It provides air to the transducer and motor air bearings. Additionally, it serves as a circulating medium for the gun heaters in the oven. It is very important that good quality air is supplied to the ARES test station at all times during use, otherwise significant damage to the instrument can result.

The optional N2/GAS input is available for using other gases as the oven-heating medium when the sample being tested would react with normal air. Nitrogen is commonly used in this case. Use the Gas Supply to Oven Selector Switch (black knob below gauges) to choose which gas input port is used to supply gas to the oven.

### Gas Pressure Specifications

Air should be supplied to the ARES at 5.5 scfm (156 l/min) at a pressure of 80 psi (5.5 bar). Gas connected through the N2/GAS port should be supplied at 60 to 70 psi (4.8 bar). The gas supplied to the various components is regulated using the Air Pressure Adjust Knobs on the backside of the panel. Pull the knob out and rotate it clockwise to increase the pressure to the desired component. Pressure is monitored using the associated gauges on the side of the Pneumatics panel.

Pressure to the various components should be adjusted as follows:

COMPONENT	REQUIRED PRESSURE
Transducer	35 psi
Motor	60 psi
Oven	40 psi (with oven on)

## Air Quality Specifications

It is critical that high quality, clean dry air is supplied to the test station at all times. Any particles present in the air must be smaller than 5 microns. Since the motor and transducer use air bearings, larger particles in the air can easily damage the delicate high precision bearing surfaces of these components. Damage to the bearing surfaces will result in faulty measurements, and if significant enough, the damage will require the replacement of the entire motor or transducer.

The relative humidity of the supplied air should be 35% to 70% with a dew point of 10 deg C. We highly recommend installing the optional air dryer/filter between the test station and the air supply. If your air supply has excessive moisture levels, which result in the immediate condensation into water, an additional water trap will be required before the air dryer.

Excessive moisture in the air supply will damage the test station.



**CAUTION: Read the operating and maintenance instructions that were supplied with your air dryer. Failure to properly operate and maintain your air dryer will result in extensive damage to this instrument.**

---

Because of the critical nature of the air supplied to the instrument, and the potential for expensive damage through mishandling, we highly recommend that you inform your laboratory manager, or compressor maintenance personnel, of your instrument requirements in detail. You should also ask to be informed before any air supply interruption, or compressor maintenance, so that you can properly shut down and protect the test station. Typically after any compressor maintenance, there will be some residual particulates and moisture present in the supply lines. You should disconnect the air from the test station input (or air dryer) and purge the lines, before re-powering your instrument.

To quickly determine if there is an interruption in the air supply, we have found that it is helpful to install a pressure gauge before the air dryer.

## Pneumatic Connections

Figure 2-15 shows the location where all the pneumatic connections are made. Connections are made using standard fittings. The connections to the air supply MAIN and N2/GAS ports use quick disconnect fittings. Table 2-3 identifies and describes Pneumatic connections (on Pneumatics Panel) necessary for test station operation.

Table 2-3. Pneumatic Connections

Connector	Destination	Purpose
AIR SUPPLY - MAIN	Connected to the (external) air dryer.	Pneumatic input to the Test Station. Used to supply air to the oven and throughout the Test Station. Supply pressure should be 80 psi.
AIR SUPPLY - N <sub>2</sub> /GAS	Connected to an external nitrogen (or other) gas supply.	Pneumatic input to the Test Station. Supplies nitrogen (or other) gas to the oven, thus allowing Nitrogen gas to be used as the heating medium. Supply pressure should be 60 to 70 psi.
GUN HEATERS	Connected to the oven.	Pneumatic output from the Test Station to the oven. Supplies the oven with air or N <sub>2</sub> gas depending upon position of Gas Supply to Oven selector switch (black knob below pressure gauges).
OVEN PRESSURE SENSOR	Connected to the oven.	Pneumatic input to the Oven Pressure Sensor, which is used to monitor oven gas pressure. The gas pressure must be above 35 psi for the oven to function.
PURGE	Connected to the oven.	Pneumatic input to the Oven that accepts air, which is circulated throughout the Oven in order to maintain positive pressure and minimize frost when using LN <sub>2</sub> .

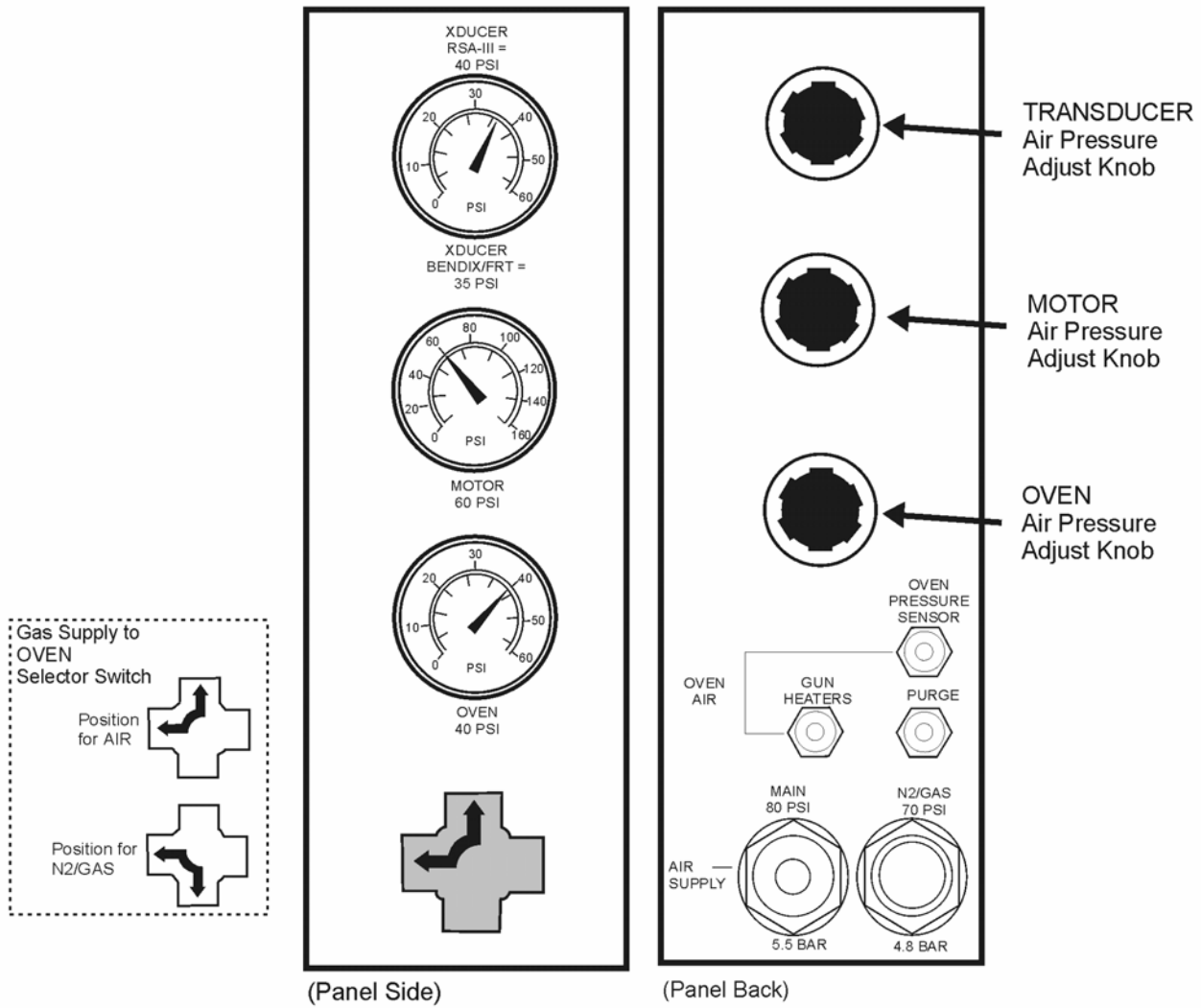


Figure 2-15. Pneumatics Panel.

This panel has 2 sides, and wraps around the right rear corner of the Test Station.

# Environmental Control Systems

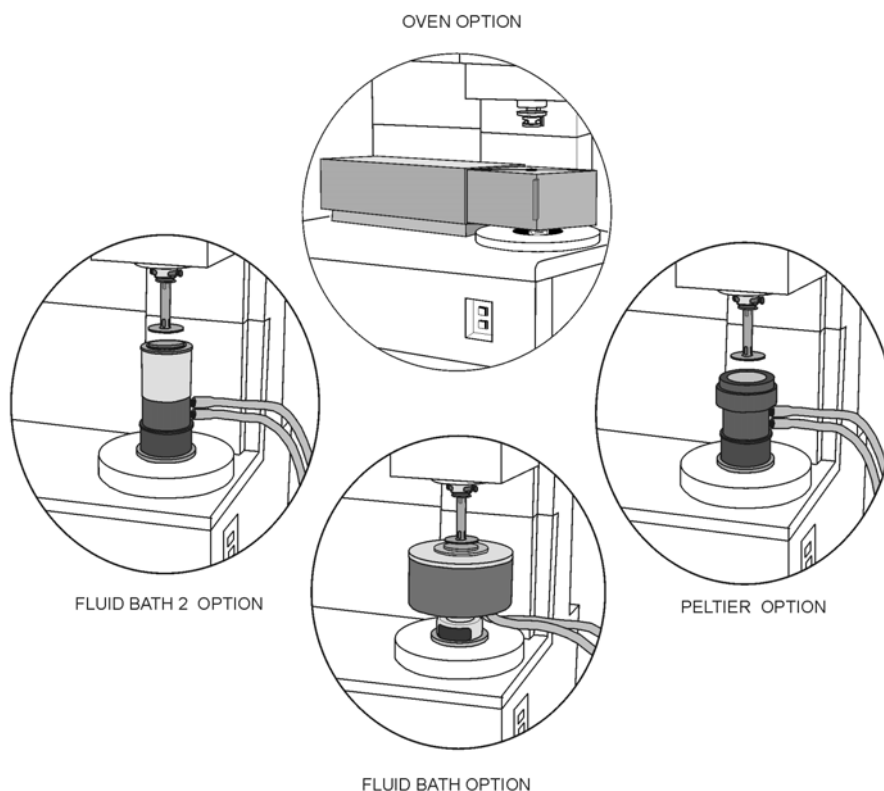
There are three environmental control systems available for use with ARES. Each system is used to precisely control sample temperature. The three systems are a forced air convection oven (Oven), a re-circulating fluid bath, and a rotating/oscillating Peltier.

The air convection oven has a dual-element heater with counter-rotating air flow for a wide temperature range (ambient to 600°C) and maximum temperature stability. If temperatures below ambient are required then either the optional LN2 controller (operation to -150°C) or the mechanical air Chiller (operation to -60°C) can be used to extend the operational range of the oven. The air convection oven is recommended for polymer melts and solids, and can accommodate Cone and Plate, Parallel Plate, Torsion Rectangular and Torsion Cylindrical geometries.

The recirculating Fluid Bath 2 (or original Fluid Bath) is appropriate for liquid samples that may evaporate under the influence of circulating air. The temperature range of the bath is approximately -20° to 140° C (depending upon the Circulator and bath fluid used), and can be used with Parallel Plates, Cone and Plate, Couette, and Double Wall Couette geometries.

Our patented rotating/oscillating Peltier system has a temperature range of -30° to 150° C, with heating rates as high as 30° C/min. The Peltier system can be used with Cone and Plate or Parallel Plate geometries.

Refer to the following sections for information regarding each specific environmental option:



## Oven

The oven is a forced air convection environmental chamber that encloses the sample. Mounted in the oven are two resistive heaters (gun heaters) that are used to control the temperature of the gas that is input to them. During testing at or above ambient temperature, either air, or nitrogen gas, can be input to the heaters. If test temperatures must be extended to below ambient, the input to the heaters must be evaporated liquid nitrogen ( $\text{LN}_2$ ) supplied by the optional cryogenic LN2 Controller or very cold gas supplied by the optional Chiller.

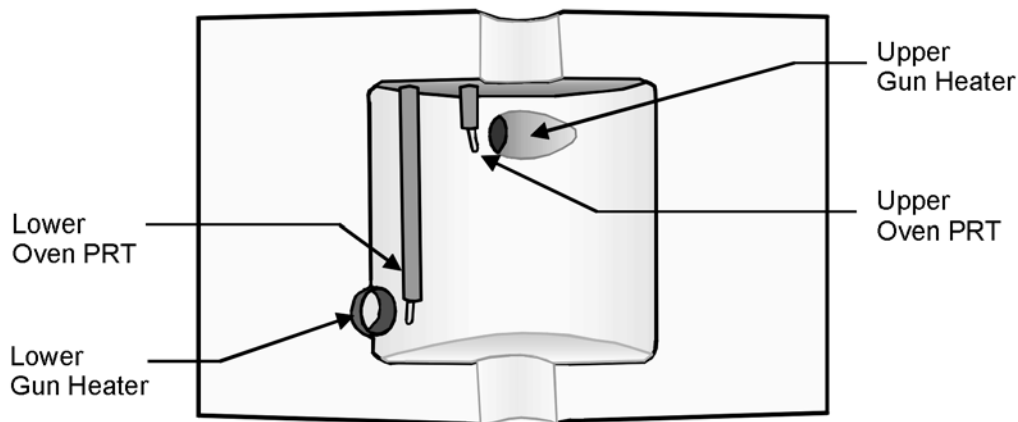


Figure 2-16. Oven Chamber Showing Gun Heaters and PRTs.

## Oven Temperature Control

Oven temperature is maintained by a control loop that is closed around a Platinum Resistance Thermometer (PRT). The ARES has two control loops (Oven PRTs) to minimize vertical temperature gradients, each of which is located in front of the respective gun heater being monitored. The control system determines actual oven temperature by supplying a constant current to the PRT and measuring the voltage developed across it. The difference between commanded and actual oven temperature is continuously driven to minimum.

You can choose to control the temperature of either the oven environment, or in some cases, the lower tool. The choice is made using Orchestrator software. The temperature of the lower tool can be monitored using the lower tool PRT, which is mounted on the motor. Installation of the lower tool PRT is covered as part of the lower tool installation in Chapter 3. Under normal operating conditions the oven PRTs are used to control oven temperature and the tool PRT is used to report sample temperature. However, the tool PRT can be used to control the oven temperature as well.

Figure 2-16 shows the inside of the oven chamber and the location of the gun heaters and two PRTs. Table 2-4 contains a complete description of temperature control loop options.

## Chiller

The Air Chiller extends the lower range of the oven to  $-60^{\circ}\text{C}$  by use of mechanical refrigeration. The Chiller is a single integrated package, consisting of air filters, air dryers, and the refrigeration unit. It is designed to connect directly to Test Stations equipped with a Forced Convection Oven. The Chiller requires a separate air input line at 85 psi and 4 scfm. Air is sent through the filter and air dryer, and then is input to the refrigeration unit where it is chilled to  $-70^{\circ}\text{C}$ . The cold air is then sent on to the oven.

### Connection

The Chiller air output connects to the Forced Convection Oven. Connect the signal cable from the Chiller to the LN2 port on the Signal Panel (Figure 2-12). Plug the Chiller into the appropriate power source. Installation should be performed by a qualified TA Instruments service technician.

### Software Configuration

The Chiller operates using Orchestrator software. The following sections discuss set up and operation of the oven, including use of either the LN2 controller or Chiller.

### Chiller Operation

To operate the Chiller turn on the Polycold power switch (located on the opposite side of the refrigeration unit as the air dryer, J-box and air filter) and J-box on/off switch (Figure 2-17). Verify that the pressure regulator gauge indicates 60 psi. Adjust the pressure regulator, if necessary, to achieve 60 psi by turning the knob clockwise to decrease the pressure and counterclockwise to increase pressure.

During extended use, parts of the Chiller may freeze inside, blocking gas flow. To help prevent damage to the gun heaters (due to insufficient gas flow), the Chiller is equipped with an emergency shut off that will shut down the compressor, and turn off the heaters, if the airflow through the Chiller drops. If this happens turn off the Chiller and allow it to thaw before continuing. Freezing will generally happen if the dew point of the input gas is greater than  $-80^{\circ}\text{C}$ . The air dryer is designed to provide dry gas to the Chiller with a dew point sufficient to prevent freezing. The Chiller should be turned off when it is not actually in use and the air dryers should be properly maintained and inspected. (Note that the air dryer is intended to dry the air supplied to the Chiller only and is not intended for any other air supplies.)

### Maintenance

There are no user-serviceable parts inside the refrigeration unit. The compressor and condenser are lifetime lubricated and do not require oiling. The condenser fins and grill should be kept clean to ensure good airflow. Clean them as needed by vacuuming or blowing with compressed air. The air filters should be inspected and cleaned as necessary. Also, any moisture should be drained from the filter housings regularly. Refer to the air dryer, air filter, and refrigeration unit documentation that came with the Chiller for further maintenance information.

The balance pressure should also be periodically monitored as described in the Polycold documentation included with your Chiller. Upon receiving the instrument, you should note and record the pressure reading on the suction gauge. This gauge is located on the opposite side of the refrigeration unit as the air dryer, air filter and J-box. The Chiller should be off for 24 hours before taking a balance pressure reading. The initial reading should be over 100 psig. Subsequent reading should be within 7 psig of the original value. If the balance pressure drops more than 7 psig, there may be a leak in the refrigeration system.

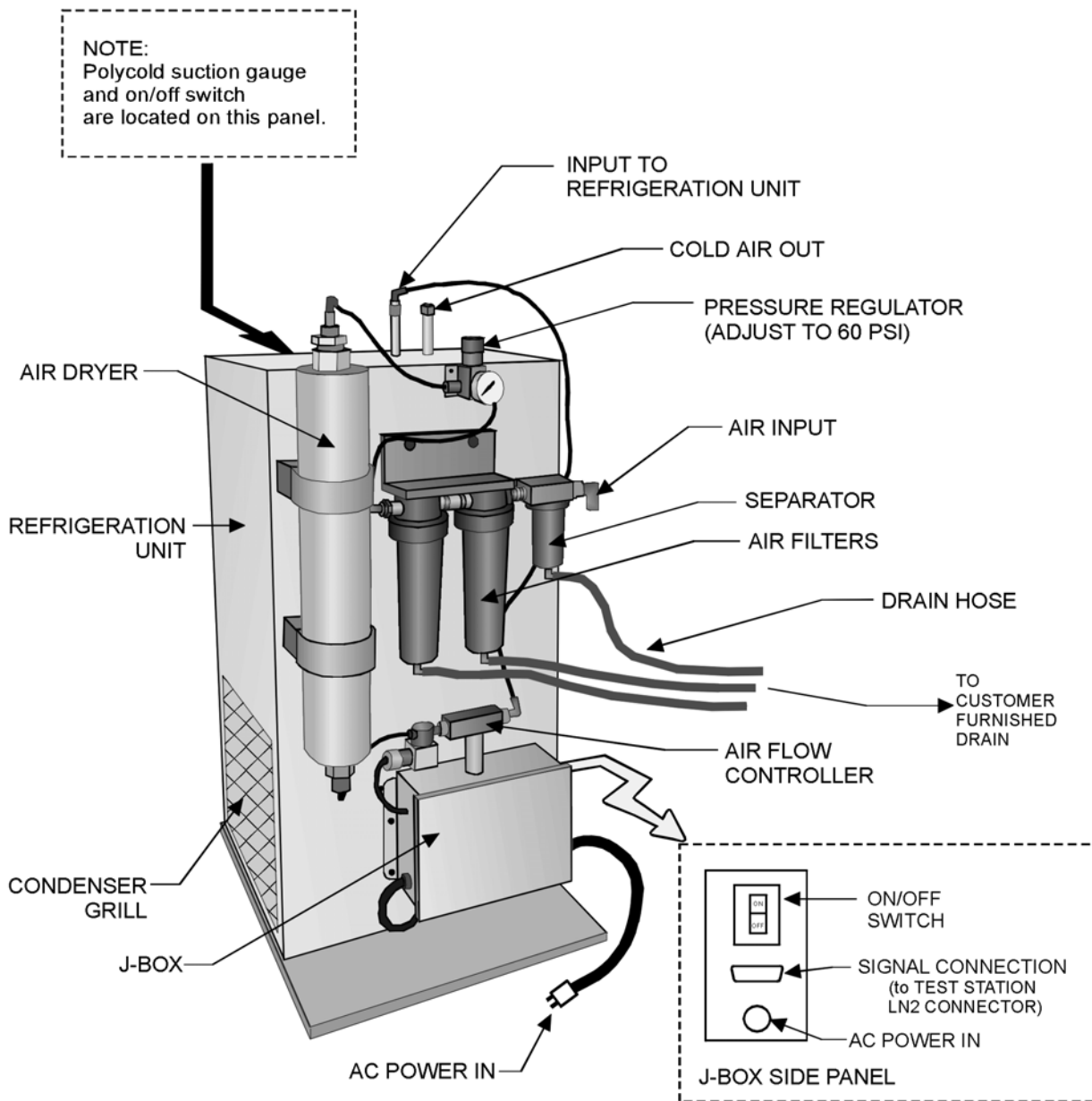


Figure 2-17. Chiller, Showing Various Components



## LN2 Controller

The LN2 controller extends the lower range of the oven to -150° C using liquid nitrogen. The LN2 controller is connected between an external liquid nitrogen source and the Test Station (Figure 2-1). Controlled by the test station through Orchestrator software, the LN2 controller consists of a Dewar flask, a solenoid valve to control liquid nitrogen flow into the Dewar, and hardware to control the boiling of the liquid nitrogen to produce very cold nitrogen gas within the flask. A second solenoid valve regulates the flow of the cold nitrogen gas out of the Dewar flask for use by the Test Station.

### Dewar Flask

A Dewar flask is a container specifically designed to efficiently store liquid nitrogen. Dewar flasks help prevent evaporation due to their double-wall construction. Within the Dewar flask, a heater immersed in the LN<sub>2</sub> provides controlled boiling. The Dewar is equipped with a pressure relief valve to prevent damage to the system. It is common for the relief valve to open (loud pop) several minutes after the LN2 controller is shut off, as the residual liquid nitrogen in the Dewar evaporates to gas.

### Connection

The LN2 output connects to the Forced Convection Oven. Connect the signal cable from the LN2 controller to the LN2 port on the Signal Panel (Figure 2-12). All connections to the ARES are normally made during system installation by a TA service technician. Connect the LN2 controller filler hose to your liquid nitrogen source. The external liquid nitrogen tank should have a low-pressure (<30 psi) valve. The hose between the nitrogen tank and the LN2 controller should be kept as short as possible to maximize the LN2 controller performance.

### Software Configuration

The LN2 operates using Orchestrator software. Chapter 2 discusses the set up and operation of the oven, including use of either the LN2 controller or Chiller.

### LN2 Controller Operation

Open the valve on the liquid nitrogen source. All remaining operation of the LN2 controller is performed through Orchestrator. In order to fill the Dewar, the LN2 Controller Dewar must be turned ON using the Orchestrator *Instrument Control Panel*. While the Dewar is filling the software system status will indicate LN2 filling, and temperature control is not active. Once the Dewar is filled, the software status will indicate LN2 READY, at which point temperature control will be active.

## Maintenance

There are no user serviceable parts inside the LN2 controller. Periodic servicing is performed in conjunction with Test Station maintenance visits by Technical Services.

### LN2 Transfer Line Maintenance

This transfer line utilizes an insulating vacuum jacket. The stainless steel used to manufacture this transfer line “outgases” under vacuum conditions. This outgasing degrades the transfer line’s vacuum jacket, and therefore its insulating properties. Absorbents or “getters” are added in the vacuum space to absorb the gases and prevent the metal’s outgasing from destroying the transfer line’s insulating vacuum.

The “getters” must be renewed periodically to maintain their absorbent qualities. This is simply done by using the transfer line to transport nitrogen gas from the dewar to the oven. The low temperature of the N<sub>2</sub> gas (-170 degree C) activates the absorbent qualities of the “getters.”

**NOTE:** Operate the LN2 controller for 15 minutes at least once every 60 days to maintain the insulating properties of the LN2 transfer line.

## Oven Gas Selection

Either gaseous nitrogen (N<sub>2</sub>) or air can be flowed through the heaters during a test. To switch between N<sub>2</sub> and air, use the Gas Supply to Oven Selector Switch, located below the pressure gauges on the Pneumatics side panel (Figure 2-15). Rotate the valve to the desired position (positions are labeled).

When using the optional LN2 Controller you can select to flow gases through the heaters in either of the following two ways through software control:

1. Use evaporated liquid nitrogen (LN<sub>2</sub>) at all times
2. Use liquid nitrogen until a specified temperature is reached, then use either nitrogen gas or air thereafter, depending upon which gas input port is selected with the Gas Supply to Oven Selector Switch.
3. When using the gas switching option, the input temperature for switching from LN<sub>2</sub> to Gas should be approximately 10 degrees higher than the input to switch from Gas back to LN<sub>2</sub>. This will prevent the system from switching back and forth between the two sources at the switch temperature since, when switching from liquid nitrogen to gas, there is a momentary change in temperature as the switch takes place.

Enable the Gas switching option, and set the switch point temperatures, using the *Set Test Conditions* function in the Control pull down menu of Orchestrator (Figure 2-18).

Figure 2-18. Set Test Conditions Form Showing Gas Switching Enabled  
 Note that N2 in the form refers to liquid nitrogen.

## Oven Operating Requirements

The oven can operate only if the following conditions are met:

- The oven is selected as the current environmental system.
- The environmental controller is turned ON in the *Instrument Control Panel*.
- The oven must be positioned all the way to the right, and the oven must be closed.
- Either air or N<sub>2</sub> must be supplied to the oven at a pressure greater than 35 psi.
- If using the LN2 Controller, the liquid nitrogen (LN2) level in the Dewar Flask must be between 50 and 75% of capacity. When it is, the Orchestrator Online Indicator **LN2 Ready** is displayed.

## Oven Configuration in Orchestrator™

When you command a temperature, Orchestrator software uses the Instrument Configuration to determine the environmental system currently in use, and establish operating conditions. Prior to operating the oven, access the *Instrument Configuration* function, located under the *Service* function of the *Utilities* pull-down menu, and set up the instrument using the guidelines shown in Figure 2-19.

The screenshot shows the 'Setup Instrument Options' dialog box with the following settings:

- Instrument Setup: Temperature Control
- Temperature Control: Oven (Air, Chiller or LN2 Dewar.)
- Maximum Temp.: 500.0 °C
- Minimum Temp.: -150.0 °C
- Cooling Temp Controller Attached:  LN2
- Auto Gas/LN2(chiller) Switching Avail.:  Yes
- Temperature Loop Control: Oven Air Temperature
- Temp Calibration Table:  Default

Figure 2-19. Setup Instrument Options Form  
Used to input the environmental system configuration.

### NOTES:

- (1) Make sure the following are selected:
  - Instrument Setup = TEMPERATURE CONTROL
  - Temperature Control = OVEN(AIR, CHILLER, OR LN2 DEWAR)
- (2) Ensure that the maximum and minimum temperatures correspond to the desired allowable operating range. Normally these are set to the instruments environmental system limits.
  - Be sure to set cooling controller to LN2, if this optional feature is installed and used.
  - Select the Temperature Loop Control option based upon Table 2-4.

Table 2-4. Oven Temperature Control Loop Options

Mode	Temperature is controlled by...	Temperature is reported by...	Temperature Control Method
1. Oven Air Temp	Oven PRTs (both upper and lower)	Tool PRT	Gas entering the Oven is maintained at the commanded temperature. Temperature is reported by the Lower Tool PRT.
2. Sample/ Tool Temp	Tool PRT	Tool PRT	Oven temperature is maintained at the commanded temperature using the Lower Tool PRT. Temperature is reported using the same PRT.
3. RAA Oven Air Temp	Oven PRTs (both upper and lower)	Lower Oven PRT	Oven gas temperature is maintained as in Mode 1. Temperature is reported using the lower Oven PRT. Designed for use when lower tool does not support a Tool PRT (such as Torsional/Rectangular test tools).
4. RAA Tool PRT	Tool PRT	Lower Oven PRT	Oven temperature is maintained as in Mode 2. Temperature is reported using the lower Oven PRT. NOT NORMALLY USED WITH ARES.

## Oven Operation

The oven is operated using the *Instrument Control Panel* (Figure 2-20). The options displayed on the *Instrument Control Panel* will be a reflection of the Environmental System Configuration entered using the *Instrument Configuration* function. Table 2-5 describes the features of each option.

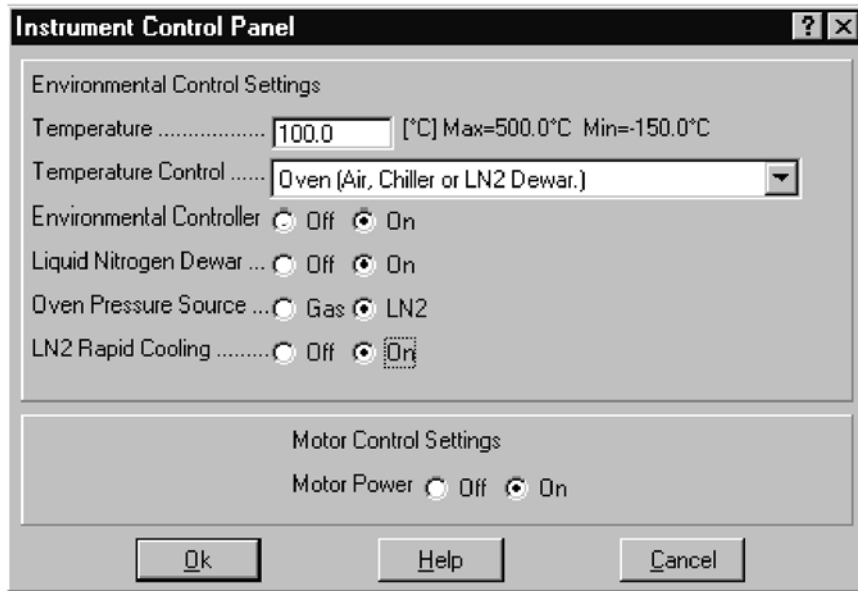


Figure 2-20. Control Panel showing Environmental Control Options

Table 2-5. Description of Oven Control Options in the Instrument Control Panel

OPTION	DESCRIPTION
Temperature	Desired temperature to conduct test (commanded temperature).
Temperature Control	Selects the environmental system (oven, bath) in use. Set this to - Oven (Air, Chiller or LN2 Dewar).
Environmental Controller	Turns on/off the oven environmental system.
Liquid Nitrogen Dewar	Turns on/off the LN2 Controller. If on, LN2 Rapid Cooling is displayed on the form.
Oven Pressure Source	Selects the gas input to the oven. Selecting Gas supplies either air or gaseous N <sub>2</sub> . Selecting LN2 supplies evaporated LN <sub>2</sub> from the LN2 Controller.
LN2 Rapid Cooling	Turns on/off rapid cooling, which evaporates LN2 at the maximum possible rate. Rapid Cooling is always on at temperatures lower than -124°C.

## Oven System Status Indicators

Two special purpose neon lamps, located on the front of the oven cover, inform you of the status of the two gun heaters. The top lamp shows the output level of the top heater. When the lamps are at their brightest, the heater guns are full ON.

Orchestrator Online Help provides instructions for choosing Online (instrument status) Indicators to be displayed along the bottom of the screen. The following indicators can be displayed, which show the status of the oven environmental system:

Indicator	Meaning
Environment On/Off/Oven Open	
Oven Open	The oven is not closed. This condition disables the oven environmental system.
Env On	The oven environmental system is currently enabled.
Env Off	The oven environmental system is currently disabled.
Gas/LN2 Status (Oven Only)	
Gas	Either air or gaseous N <sub>2</sub> is currently input to the oven.
LN2 Fill	The LN2 Controller Dewar flask is currently filling with liquid nitrogen, and the LN <sub>2</sub> level is less than 50% of the flask capacity. Temperature control will not be active while the Dewar flask is filling.
LN2 Ready	The LN2 Controller is currently ready for use. In this case, the LN <sub>2</sub> level in the Dewar flask is between 50 and 75% of the flask capacity.
LN2 Off	The LN2 Controller is currently disabled.

# Fluid Bath 2

## Description

The Fluid Bath 2 offers precise control of sample temperature using a closed fluid re-circulant system. The operational range of the Fluid Bath 2 is  $-10^{\circ}\text{C}$  to  $140^{\circ}\text{C}$ . Thermally controlled fluid, supplied by a circulator, flows through the bath. The lower test tool is mounted within the Bath Well, and attains thermal equilibrium with the surrounding bath. The temperature of the lower tool is measured by the bath PRT, which mounts through the Bath Well into the Motor. You can choose to control the temperature of either the lower tool or the circulator fluid itself.

The Circulator regulates the temperature of the bath fluid, and pumps the fluid through the Fluid Bath. The circulator (as supplied by TA Instruments), is connected to, and is under the control of, the test station and software. The fluid circulated through the bath is maintained at the temperature selected in Orchestrator. The circulator also has its own fluid temperature regulation, which can be used as the temperature control loop for the Bath by selecting manual temperature control Orchestrator. Presently, the standard circulator supplied by TA Instruments is the Julabo FS-18.

## Installation of Fluid Bath

The fluid bath is mounted onto the motor using a threaded collar.

Three screws fasten the rotating Shaft of the Fluid Bath (Figure 2-21) to the Test Station Motor Anvil. A threaded collar secures the Fluid Bath Body to the Test Station Motor Housing.

Two hoses supply fluid between the Fluid Bath and the fluid source, which is typically a computer-controlled circulator.

Prior to mounting the Fluid Bath, perform the following actions on the ARES instrument:

- Raise the Stage to maximum height.
- Remove all Upper and Lower Test Tools, and loosen the Anvil Tightening Knob on the Motor Anvil.
- Thoroughly inspect the Test Tool mounting surfaces, i.e., the transducer anvil and the motor anvil and clean off any material that may interfere with the mounting of the Fluid Bath. This is essential to ensure proper mechanical mating between the bath and the instrument.
- Turn off the Motor, using the *Instrument Control Panel*.

Figure 2-22 while performing the following steps to install the bath:

1. Remove the protective plastic base from the Fluid Bath Threaded Collar by placing the two pins on the Threaded Collar Spanner Wrench (provided) into two of the holes machined into the Collar, and rotating the wrench counterclockwise.
2. Gain access to the Shaft by sliding the cover of the Fluid Bath fully upward.
3. Hold the Fluid Bath above the ARES Motor Anvil with the Bath Hoses facing toward the right of the instrument.

4. Rotate the Fluid Bath Shaft to align the flat portion of the Shaft with the flat portion of the ARES Motor Anvil (both flats should be facing toward the right as you face the instrument).
5. Slowly lower the Bath onto the ARES Motor Housing, ensuring that:
  - The three screws in the Shaft align with the three threaded holes machined into the ARES motor anvil.
  - The Pin (Figure 2-23) machined into the bottom of the Fluid Bath is seated into the notch in the ARES Motor Housing (the Pin and notch should be located toward the rear of the instrument). It may be necessary to rotate the bath back and forth until the Pin falls into the notch.
  - The Threaded Collar of the Fluid Bath rests in the threaded portion of the ARES Motor Housing.
  - Ensure that the Spring (Figure 2-21) is positioned behind the Anvil Tightening Knob on the ARES Motor. Rotate the Motor Anvil to gain access to the Anvil tightening knob then tighten the knob. A flat-head screwdriver may be used if you do not overtighten.

While the next steps are listed sequentially, they should be, in practice, performed more or less simultaneously. That is, the collar should be tightened a small amount, then screws started, then the collar some more, to ensure that all components are aligned and mating properly. If at any time a part does not seem to fit well, or tighten easily, stop and ascertain the reason for the problem. Do not force anything.

1. Tighten the Threaded Collar by placing the two pins on the spanner wrench (provided) into two of the holes machined into the Collar, and rotating the wrench clockwise. Do not overtighten the Collar.
2. Tighten the three screws in the Shaft using the supplied 5.5 mm open-end wrench. Again, do not overtighten the screws.

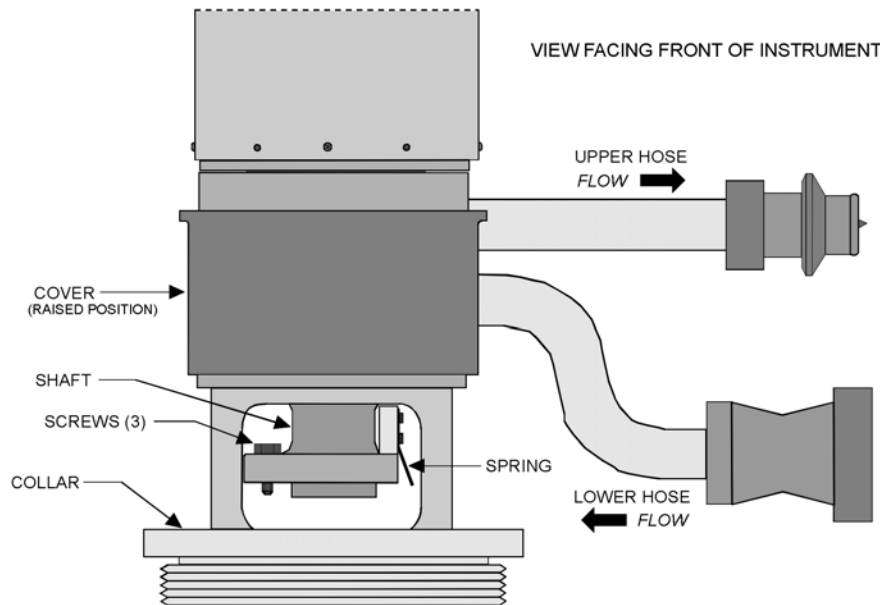


Figure 2-21. Fluid Bath 2 Lower Portion Details



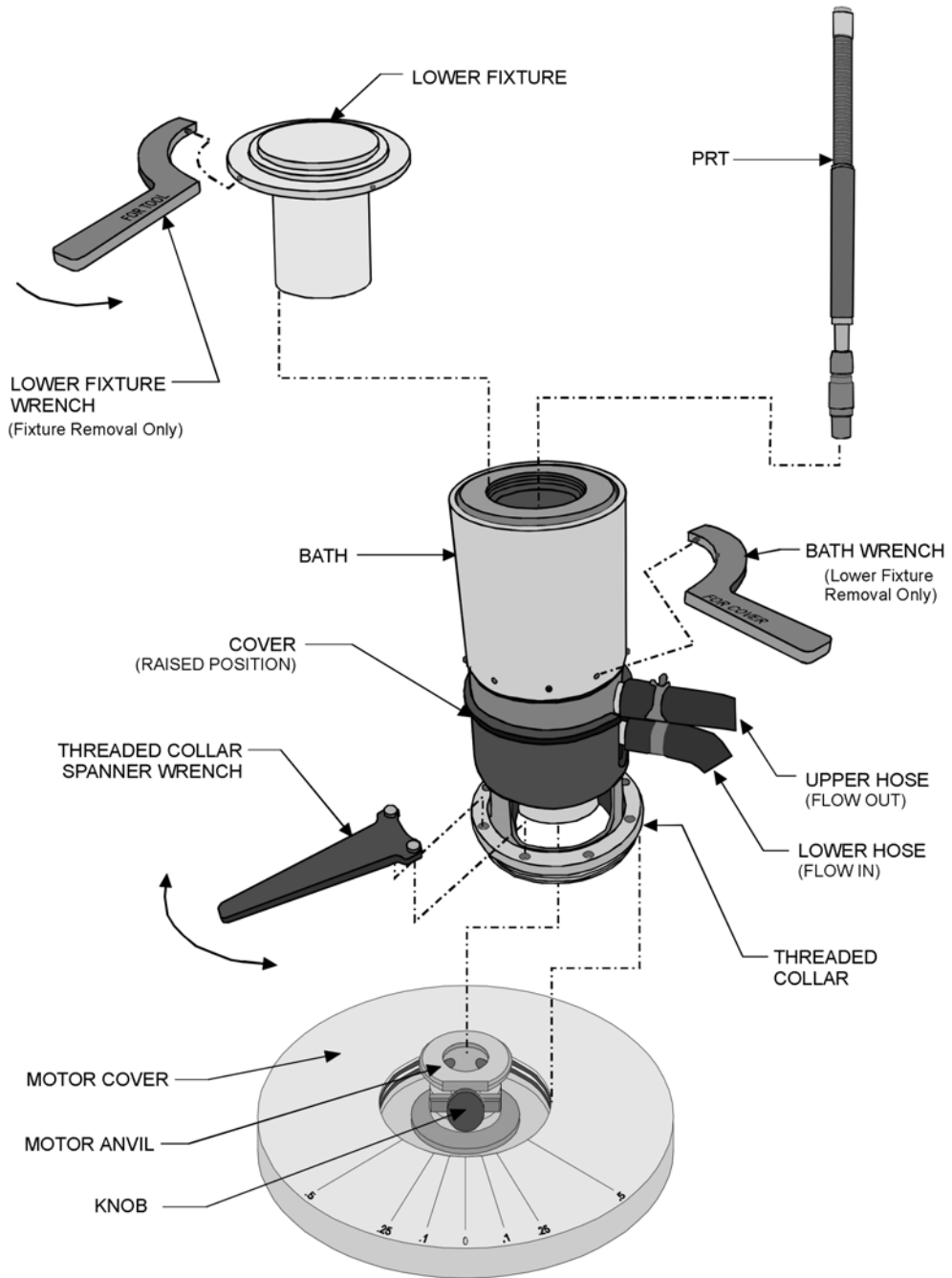


Figure 2-22. Fluid Bath 2 Components and Installation

## Circulator Connections

Connect the circulator to the bath using the supplied connectors. The lower hose on the bath is for flow into the bath (out of the circulator) and the upper hose is for flow out of the bath (Figure 2-21). The circulator should be positioned on the floor below the work area. Make sure all hoses are installed completely onto their respective hose barbs and that the hose clamps are tight. Connect cable 707-00750 between the Circulator RS-232 connector, and the Test Station Signal Panel CIRCULATOR connector.

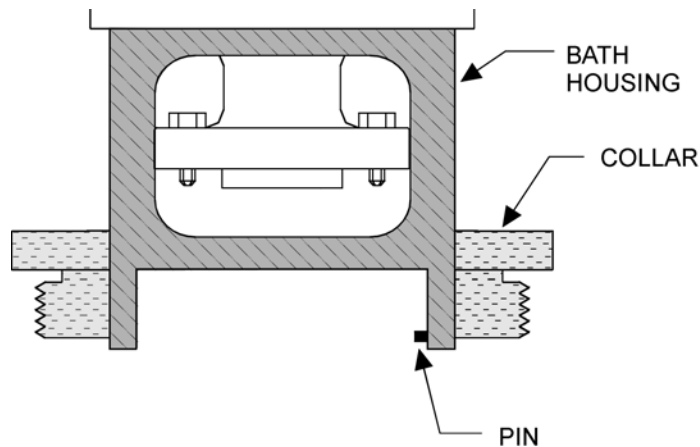
## PRT Installation

There are two PRTs available (Figure 2-24), each designed to be used with a specific lower tool. Each PRT is designed to contact the lower tool, and provides the temperature of that tool.

Note that the PRT used with the different Couettes is shorter than the PRT used with the Parallel Plate tool. Because of its length, the Couette PRT can be difficult to remove by hand. An extraction tool is provided to aid in the removal of this PRT. When removing the short PRT, slip the tool over the PRT end and engage the slot in the tool over the pin on the PRT as shown the detail of Figure 2-24. Pull the PRT straight up and out of the bath. DO NOT rotate the PRT in its mount since damage to the connector can result.

Install the lower tool PRT as follows:

1. Ensure that the proper PRT is selected from the lower tool PRTs available for use with the ARES Fluid Bath 2.
2. Using very little force, place the PRT plug (keyed end) through the hole in the bath, and onto the receptacle that is installed in the motor.
3. Gently push down on the PRT and rotate it until the PRT slips into the receptacle (the keyed end of the PRT then slips into the key slot machined into the receptacle). Seat the PRT completely into the receptacle. When properly installed, the displayed temperature parameter should indicate ambient temperature.



(CROSS SECTION SHOWN FOR VISIBILITY)

Figure 2-23. Fluid Bath Alignment Pin

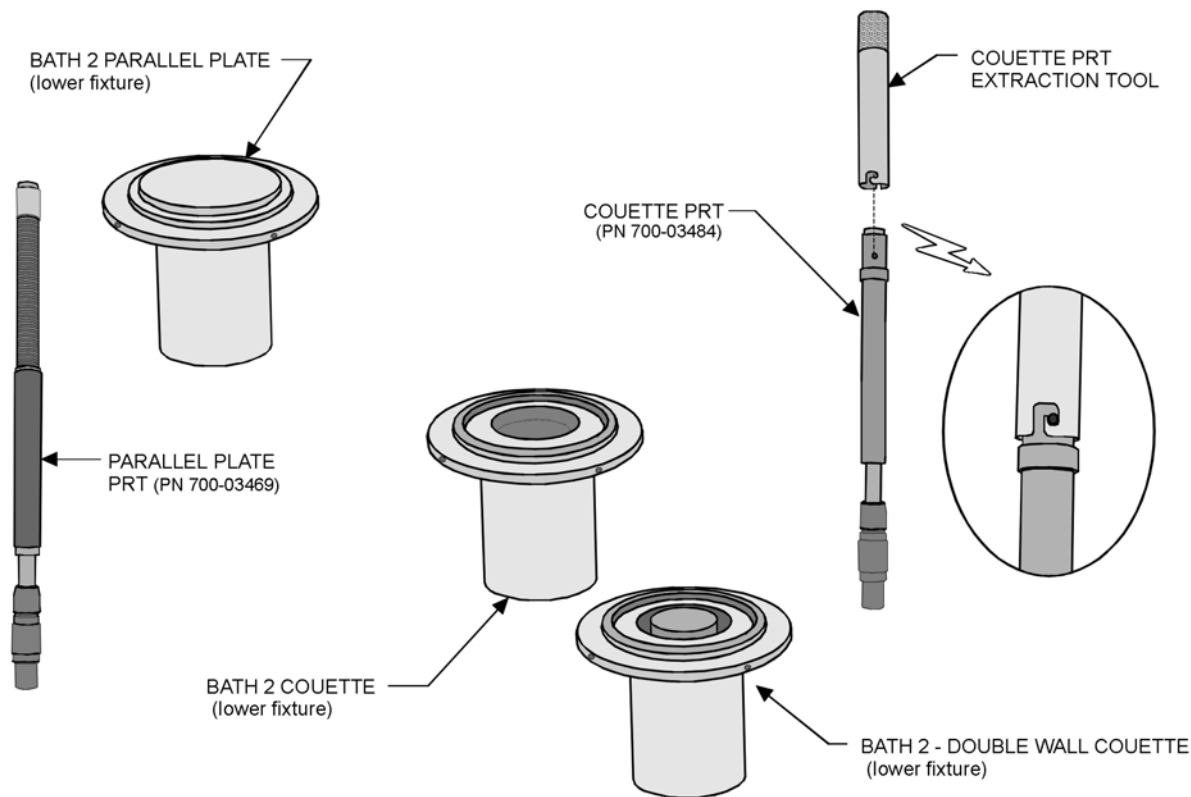


Figure 2-24. Fluid Bath 2 Lower Tool and PRT options.

## Lower Tool Installation and Removal

The lower tool is mounted into the bath well. The well is threaded to mate with the threads on the tool. The tool should be tightened clockwise, by hand only. Before installing the lower tool ensure that the correct PRT is installed and operational.

To remove the tool, use the supplied wrenches, if necessary, to loosen the tool from the bath well. The wrench used on the tool is labeled "FOR TOOL" and the wrench used to hold the bath is labeled "FOR COVER." Keeping the lower tool and bath well threads clean, and free of damage, will help ensure easy installation and removal.

## Filling the Circulator

Depending on the type of circulator in use and the desired operating range of the circulator, fill the circulator with fluid as specified in Table 2-6. The Julabo FS-18 circulator requires about 2 liters of fluid, and should be filled to within 5 mm from the top. However, refer to your actual Bath documentation for specific circulator filling and operating guidelines, as well as other bath fluid options for your application. Also refer to the bath fluid MSD for guidelines regarding the safe handling of your particular bath fluid.

Because of the construction of the Fluid Bath 2, the seals are very delicate. It is imperative that a mixture of 50% ethylene glycol / 50% water be used in the bath. Alternatively, Julabo® Thermal H10S can be used. **Using pure water is not recommended, as it will significantly reduce the lifetime of the seals.** In the event of a seal failure, the bath **must** be returned to TA Instruments for service and repair.

Table 2-6. Circulator Operating Ranges using Various Bath Fluids

Desired Operating Range of Circulator (bath temperature range is slightly less)	Fluid
-5°C to +100°C	50% ethylene glycol / 50% water
+40°C to +110°C	100% ethylene glycol
-20°C to +150°C	Julabo® Thermal H10S

## Fluid Bath 2 Configuration in Orchestrator™

When you command a temperature, Orchestrator software uses the Instrument Configuration to determine the environmental system currently in use, and establish operating conditions. Prior to operating the fluid bath, access the *Instrument Configuration* function, located under the *Service* function of the Utilities menu, and set up the instrument using the guidelines shown in Figure 2-25.

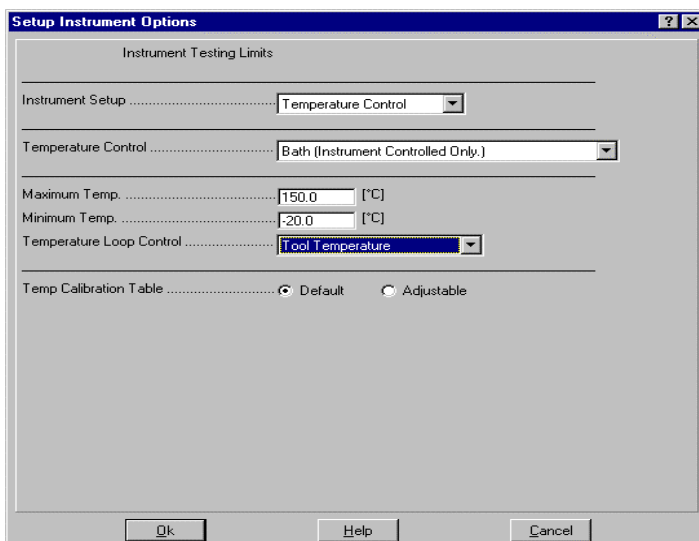


Figure 2-25. Setup Instrument Options Form Used to Input the Fluid Bath System Configuration

### NOTES:

- (1) Make sure the following are selected:
- Instrument Setup = TEMPERATURE CONTROL
  - Temperature Control = BATH (INSTRUMENT CONTROLLED ONLY)

NOTES (continued):

- (2) Ensure that the maximum and minimum temperatures corresponding to the desired circulator operating range and fluid (see the previous topic *Filling the Circulator*) are entered in the Max and Min Temperature fields.
- (3) Select either Temperature Loop Control option, using the following information as a guideline:
  - **Circulator Temperature:** When selected, the temperature of the fluid *in the circulator* is maintained at the commanded temperature. The lower tool PRT is independent of the circulator temperature, and due to its location, will report temperatures that are slightly different than the commanded (circulator) temperature. The circulator temperature control should be used when running step or ramp type temperature studies where a controlled, steady, temperature change, or speed, is more critical than actual sample temperature.
  - **Tool Temperature:** When selected, the temperature of the lower test tool is maintained at the commanded temperature. Tool Temperature control is the recommended setting for isothermal (and some step) testing, where controlling the actual sample temperature is most important. When the commanded temperature has been achieved, wait 20 to 30 minutes at the commanded temperature before starting the test to allow the bath and sample to stabilize.
- (4) For normal applications, set the Temperature Calibration Table to Default. For critical temperature work, the adjustable option can be used to enter calibration "offsets" for specific temperature "setpoints." Refer to the Orchestrator online help for details on how to set up this feature.

## PID Loop Setup

When using Tool Temperature control, errors between commanded and actual temperature are driven to zero by a PID (Proportional + Integral + Derivative) loop control system. The PID loop settings (Figure 2-26) affect how the bath settles at a set-point temperature. The values are affected by circulator fluid used, as well as the circulator specifications. As the bath reaches a setpoint there will typically be a few degrees of overshoot, after which the temperature will oscillate a few times and then stabilize. Typical stabilization times are 15 minutes to reach 0.1° C and 35 minutes to reach 0.01° C. By modifying the Proportional Band (P coefficient) less overshoot can be achieved, but there will typically be more oscillations before the bath stabilizes, so the overall stabilization time will be longer.

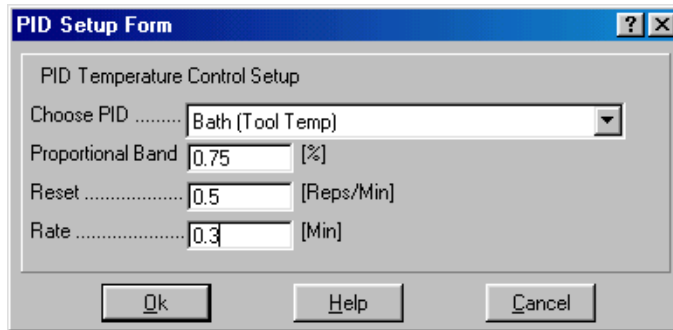


Figure 2-26. PID Setup Form

The values listed in Table 2-7 should be used as guidelines, and will work for most applications. However, you may need to experiment somewhat to determine their baths best PID settings for their system, or specific applications. The Orchestrator ON Line Help has a complete description of how to determine and tune the PID coefficients.

Table 2-7. PID Values for the Julabo® Circulator and Selected Fluids

Circulator	Julabo® FS-18
Bath Fluid	50% Water 50% Ethylene Glycol
Proportional Band	0.75
Reset	0.25
Rate	0.67

## Fluid Bath 2 Operating Requirements

The Fluid Bath can operate only if the following conditions are met:

- The Fluid Bath is selected as the current environmental system.
- The circulator must be filled, on, and circulating fluid through the bath.
- The circulator must be connected to the test station via the correct RS-232 cable.

## Fluid Bath 2 Operation

The Fluid Bath is operated using the Instrument Control Panel (Figure 2-27). The desired temperature is set in the Temperature input field. For most applications, especially isothermal testing, or when the actual tool temperature is required, Tool Temperature control is used. For temperature ramp tests, where a steady ramp rate is most important, the circulator control gives better results. If using manual temperature control for isothermal testing, you may have to adjust the circulator fluid temperature somewhat to give the desired temperature at the tool.

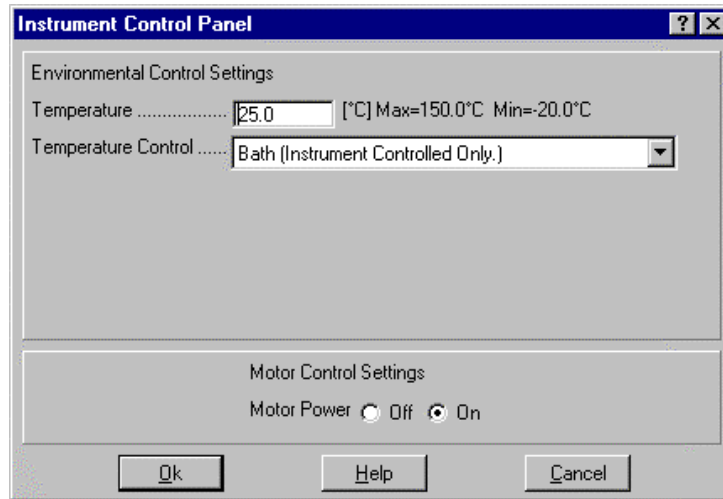


Figure 2-27. Instrument Control Panel Showing Fluid Bath Control Options

# Peltier

## Description

The Peltier environmental control system utilizes a solid-state heat pump (thermopile) to control the temperature of the lower tool, which is an integral part of the Peltier Assembly. In general, the solid-state heat pump consists of multiple semiconductor devices in series, alternating P-type and N-type. The devices are placed between the lower tool and a heat sink (Figure 2-28).

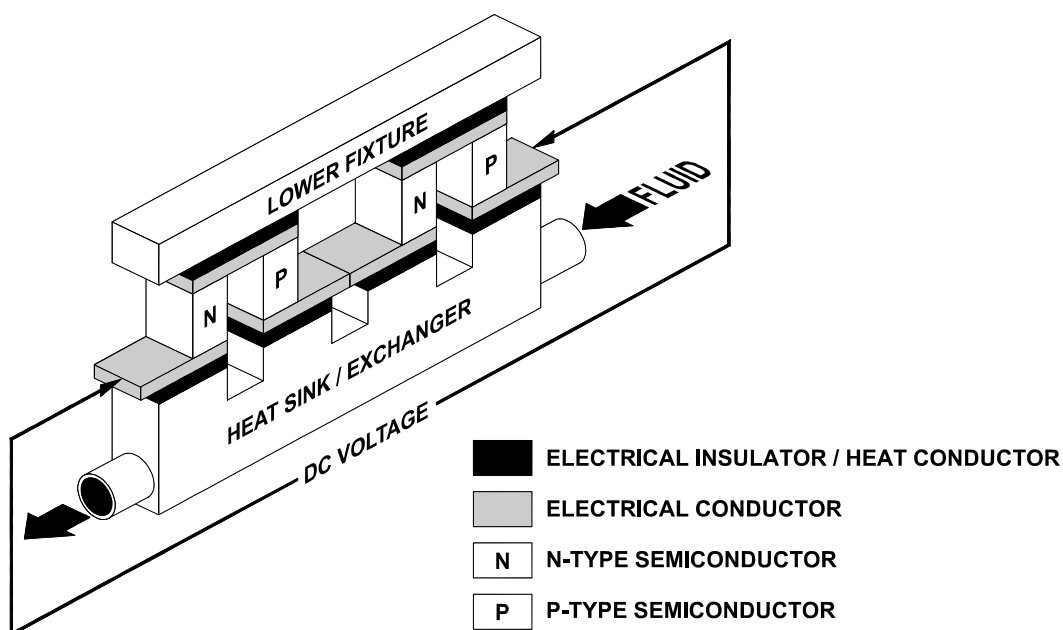


Figure 2-28. Solid State Heat Pump (Peltier Element) Schematic

DC voltage is applied across the semiconductors to either heat or cool the lower tool, depending on the polarity of the voltage. The semiconductors transfer thermal energy between the lower tool and the heat sink, which is essentially a heat exchanger through which fluid (typically water) is circulated. The fluid temperature determines the amount of thermal energy that the heat sink can source to or sink from the lower tool. The fluid temperature thereby modifies the actual operational temperature range within the overall System Specifications (Table 2-8).

Table 2-8. Peltier System Specifications.

Temperature Range	-30°C to +150°C, depending on circulator fluid temperature.
Ramp Rate	30°C per minute from 0°C to +100°C (at 20°C circulator fluid temperature)
Stability	±0.1°C



## Circulator Options

The Peltier must be connected to a circulating fluid source. Any manually controllable circulator with heating/cooling capabilities can be used to operate the Peltier system over its entire specified temperature range. This can also be the same circulator used with the Fluid Bath. The standard fittings installed onto the ARES Peltier Assembly fluid hoses accommodate computer-controlled circulators that are sold by TA Instruments.

The Peltier Circulator, manufactured by TA Instruments, is an effective yet inexpensive means of providing non-temperature-controlled circulating fluid. Instructions for use of the Peltier Circulator are printed on the label that is affixed to the circulator. If using the Peltier Circulator, you must remove the standard fluid fittings from the ARES Peltier Assembly fluid hoses, then install the special pair of fittings that are supplied for use with the Peltier Circulator

## Selecting a Thermal Operating Range

The temperature of the circulating fluid determines the Thermal Operating Range of the Peltier system. To select a thermal operating range, fluid temperature must be set in accordance with the following guideline.

Assuming that the ambient temperature in the vicinity of the heat sink is  $+20^{\circ}\text{C}$ , a low-end differential ( $\Delta T_L$ ) of approximately  $-40^{\circ}\text{C}$  exists between the fluid temperature and the lower limit of the thermal operating range. A high end differential ( $\Delta T_U$ ) of approximately  $+80^{\circ}\text{C}$  exists between the fluid temperature and the upper limit of the thermal operating range.

To determine thermal operating range for a given fluid temperature, add these differentials to the fluid temperature. For example, at a fluid temperature of  $+20^{\circ}\text{C}$ , the thermal operating range of the Peltier system is approximately  $-20^{\circ}\text{C}$  to  $+100^{\circ}\text{C}$ , calculated as follows:

Lower Limit	Upper Limit
$= \Delta T_L + \text{Fluid Temp}$	$= \Delta T_U + \text{Fluid Temp}$
$= -40^{\circ}\text{C} + (+20^{\circ}\text{C})$	$= +80^{\circ}\text{C} + (+20^{\circ}\text{C})$
$= -20^{\circ}\text{C}$	$= +100^{\circ}\text{C}$

At a fluid temperature of  $+40^{\circ}\text{C}$ , the thermal operating range of the Peltier system is approximately  $0^{\circ}\text{C}$  to  $+120^{\circ}\text{C}$ , calculated as follows:

Lower Limit	Upper Limit
$= \Delta T_L + \text{Fluid Temp}$	$= \Delta T_U + \text{Fluid Temp}$
$= -40^{\circ}\text{C} + (+40^{\circ}\text{C})$	$= +80^{\circ}\text{C} + (+40^{\circ}\text{C})$
$= 0^{\circ}\text{C}$	$= +120^{\circ}\text{C}$

## Installation of Peltier

Three screws fasten the rotating Shaft of the Peltier Assembly (Figure 2-29) to the Test Station Motor Anvil. A threaded collar secures the Peltier Assembly Body to the Test Station Motor Housing.

Electrical contact to the Test Station is established by the PRT Plug, a seven-pin male Lemo® plug that is attached to the rotating Shaft of the Peltier Assembly. During mounting of the Peltier Assembly, the PRT Plug is automatically pushed downward into the PRT Receptacle at the center of the Motor Anvil.

Two hoses supply fluid between the Peltier Assembly and the fluid source, which is typically a temperature-controlled circulator.

Prior to mounting the Peltier Assembly, perform the following actions on the ARES instrument:

- Raise the Stage to maximum height.
- Remove all Upper and Lower Test Tools, and loosen the Anvil Tightening Knob on the Motor Anvil.
- Thoroughly inspect the Test Tool mounting surfaces, *i.e.*, inspect the transducer anvil and the motor anvil and clean off any material that may interfere with the mounting of the Peltier. This is essential to ensure proper mechanical mating between the Peltier Assembly and the instrument.
- Turn off the Motor.

Refer to Figure 2-29 during the following installation procedures.

1. Remove the protective plastic base from the Peltier Assembly Collar by placing the two pins on the spanner wrench (provided) into two of the holes machined into the Collar, and rotating the wrench counterclockwise.
2. Gain access to the Peltier Assembly Shaft by sliding the cover of the Peltier Assembly fully upward.
3. Hold the Peltier Assembly above the ARES Motor Anvil with the Bath Hoses facing toward the right of the instrument.
4. Rotate the Peltier Assembly Shaft to align the flat portion of the Shaft with the flat portion of the ARES Motor Anvil (both flats should be facing toward the right as you face the instrument). At this time, the red dot on the PRT Plug should be facing toward the front of the instrument.



**CAUTION:** As you lower the Peltier Assembly in the next step, the Peltier Assembly PRT Plug will be inserted into the ARES PRT Receptacle. Do not use excessive force if resistance is encountered while lowering, as this may damage the PRT Plug and Receptacle. In this case, raise the Peltier Assembly, check the alignment described in the next step, and try again.

---

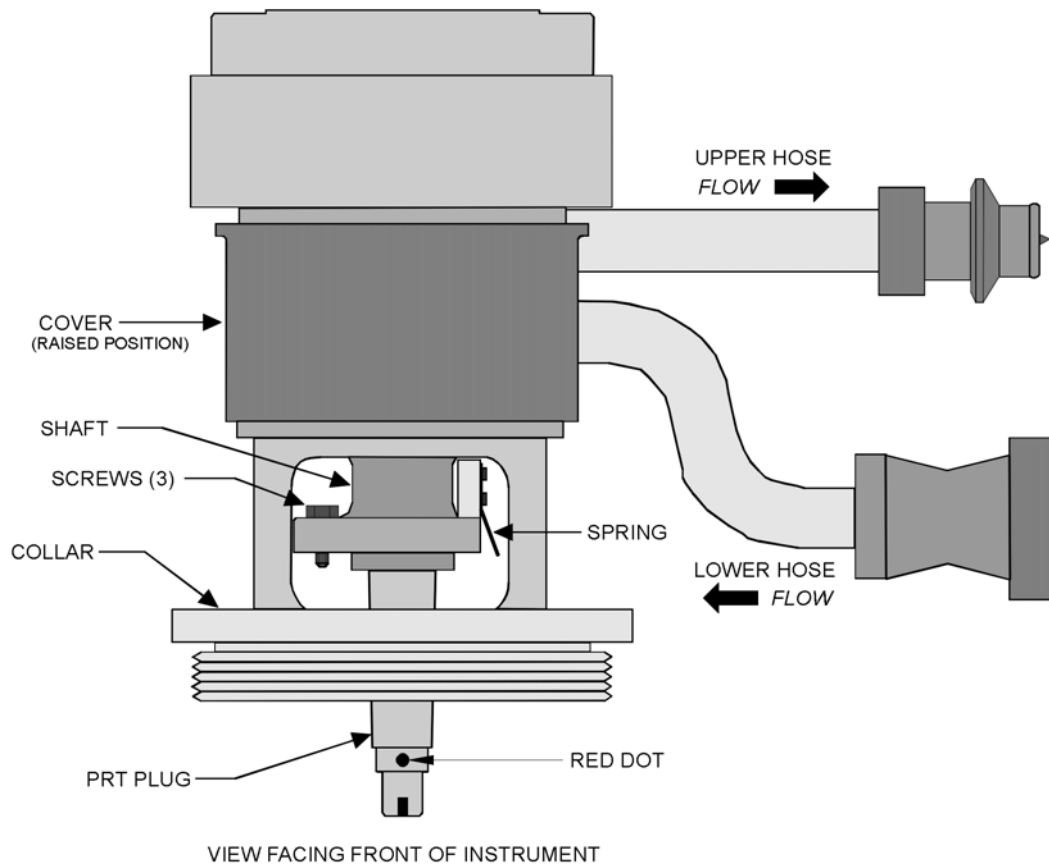


Figure 2-29. ARES Peltier Assembly

5. Slowly lower the Peltier Assembly onto the ARES Motor Housing, ensuring that:
  - The three screws in the Peltier Assembly Shaft align with the three threaded holes machined into the ARES motor anvil.
  - The Pin (Figure 2-30) machined into the bottom of the Peltier Assembly is seated into the notch in the ARES Motor Housing (the Pin and notch should be located toward the rear of the instrument). It may be necessary to rotate the Peltier Assembly back and forth until the Pin falls into the notch.
  - The threaded Collar of the Peltier Assembly rests in the threaded portion of the ARES Motor Housing.
  - Ensure that the spring (Figure 2-29) is positioned behind the Anvil Tightening Knob on the ARES Motor. Rotate the Motor Anvil to gain access to the Anvil tightening knob then tighten the knob (a flat-head screwdriver may be used if you do not overtighten).

While the next steps are listed sequentially, they should be performed more or less simultaneously. That is, the collar should be tightened a small amount, then the screws started, then the collar some more, to ensure that all components are aligned and mating properly. If at any time a part does not seem to fit well, or tighten easily, stop and ascertain the reason for the problem. Do not force anything.

6. Tighten the Peltier Assembly Collar by placing the two pins on the spanner wrench (provided) into two of the holes machined into the Collar, and rotating the wrench clockwise until the Collar is snug (do not overtighten).
7. Tighten the three screws in the Peltier Assembly Shaft using the supplied 5.5 mm open-end wrench (do not overtighten).

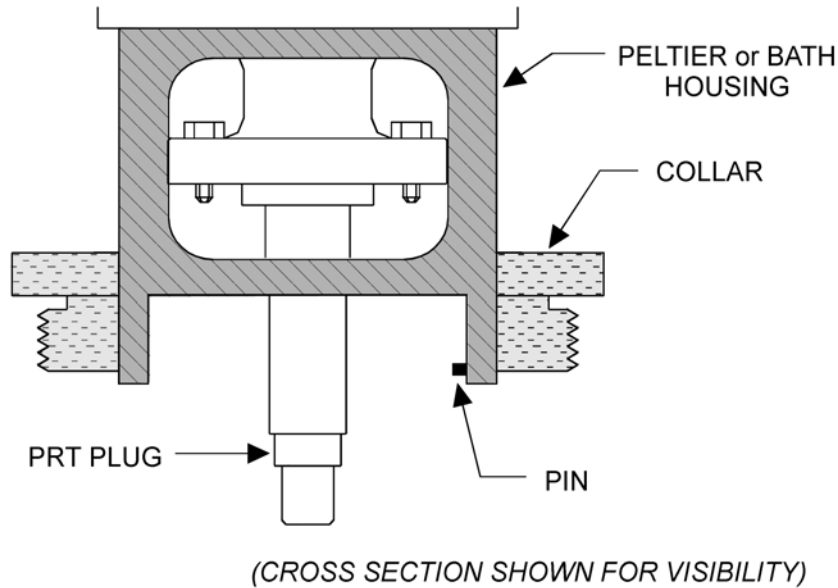


Figure 2-30. Peltier Alignment Pin

## Circulator Connections

Connect the Circulator hoses to the Peltier Assembly as follows:

Connect...	To...
Fluid Output from Circulator	Lower Hose of Peltier Assembly
Fluid Input to Circulator	Upper Hose of Peltier Assembly

The circulator should be positioned on the floor below the work area. Make sure all hoses are installed completely onto their respective hose barbs and that the hose clamps are tight.

## *Filling the Circulator*

Regardless of the type of circulator used, we recommend that the fluid used be a mixture of 50% water and 50% ethylene glycol. Use of pure water or any other mixture will ultimately result in damage to the Peltier Assembly; specifically seal degradation and corrosion.

## Peltier Configuration in Orchestrator™

When you command a temperature, Orchestrator software uses the Instrument Configuration to determine the environmental system currently in use, and establish operating conditions. Prior to operating the fluid bath, access the *Instrument Configuration* function, located under the *Service* function of the Utilities menu, and set up the instrument using the guidelines shown in Figure 2-31.

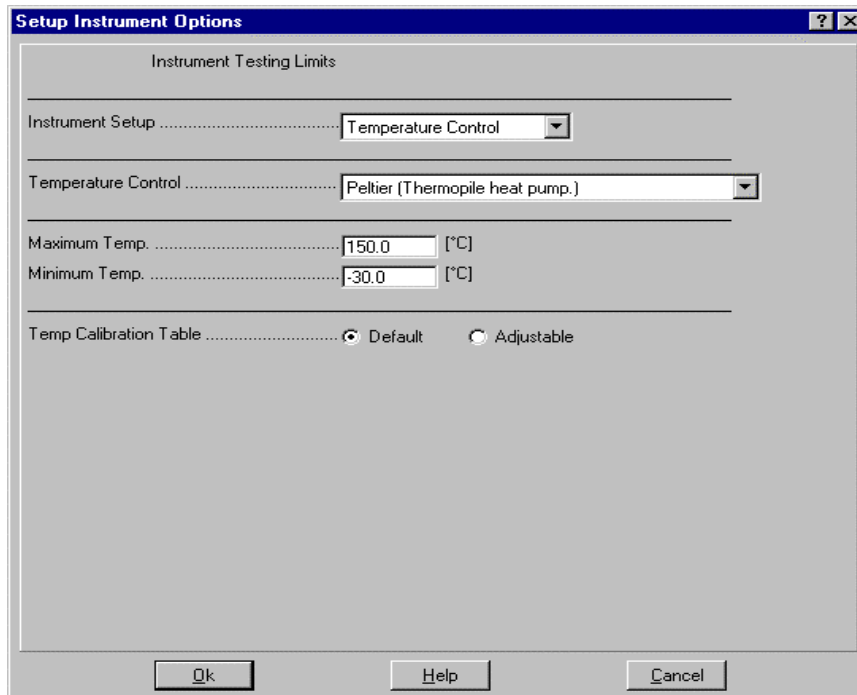


Figure 2-31. Setup Instrument Options form used to input the Peltier system configuration.

### NOTES:

- (1) Make sure the following are selected
  - Instrument Setup = TEMPERATURE CONTROL
  - Temperature Control = PELTIER (THERMOPILE HEAT PUMP)
- (2) Ensure that the maximum and minimum temperatures are set as follows:
  - Maximum Temp = +150°C
  - Minimum Temp = -30°C
- (3) For normal applications set the Temperature Calibration Table to Default.
- (4) For critical temperature work, the adjustable option can be used to enter calibration "offsets" for specific temperature "setpoints." Refer to the Orchestrator online help for details on how to set up this feature.

## PID Loop Setup

When using environmental control systems, errors between commanded and actual temperature are driven to zero by a PID (Proportional + Integral + Derivative) loop control system. The PID loop settings (Figure 2-32) affect how the Peltier settles at a set-point temperature. As the Peltier reaches a setpoint there will typically be a few degrees of overshoot, after which the temperature will oscillate a few times and then stabilize. The Peltier will typically reach stabilization within a few minutes. By modifying the Proportional Band (P coefficient) less overshoot can be achieved, but there will typically be more oscillations before the bath stabilizes, so the overall stabilization time will be longer.

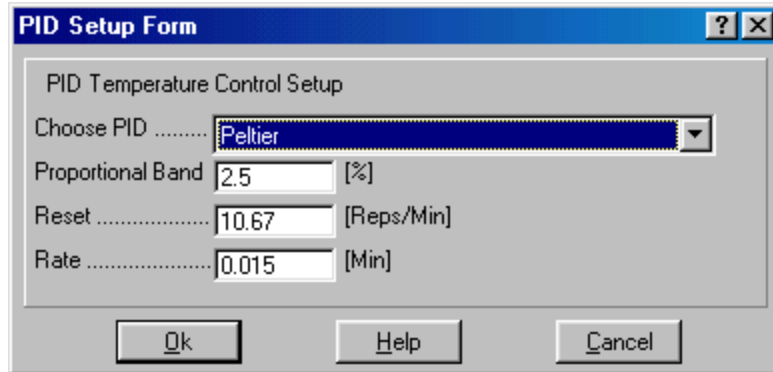


Figure 2-32. PID Setup Form Showing Peltier Settings

Table 2-9. Default PID Loop Values for Various ARES Firmware Versions

PID Loop	Version 4 Firmware	Version 5 and 6 Firmware
<b>Proportional Band</b>	.5%	2.5%
<b>Reset</b>	10.67reps/min	10.67reps/min
<b>Rate</b>	.015 min.	.015

## Peltier Operating Requirements

The Peltier can operate only if the following conditions are met:

- The Peltier is selected as the current environmental system.
- The environmental system is turned ON in the *Instrument Control Panel*.
- Fluid must be flowing through the Peltier.



**WARNING:** Do not operate the Peltier system unless fluid is being pumped through it. At all times while operating the Peltier system, ensure that the circulator is connected to the Peltier Assembly, and the circulator pump is ON. Failure to heed this warning will result in rapid evaporation of fluid trapped in the Peltier Assembly hoses, causing hose rupture, damage to the Peltier Assembly, and possible personal injury.

---

## Peltier Operation

Turn on the Circulator and set it to the desired temperature. The Peltier is operated using the Instrument Control Panel (Figure 2-33). Set the environmental control option to On. The desired temperature is set in the Temperature input field.

### Humidity Cover

After you have loaded the sample, you may install the two-piece Humidity Cover (Figure 2-34) by simply placing each half of the Cover onto the top of the Peltier Assembly and pressing the halves together. The Humidity Cover helps to create a thermally isolated chamber, thus insuring that the Peltier specifications are met. It also prevents sample evaporation during testing. The "pads" on the inside of the cover should be saturated with sample before each use to minimize evaporation.



**WARNING:** During and after testing at other than ambient temperature, the sample test surface can reach temperatures that cause personal injury. Allow the Peltier System to return to ambient temperature BEFORE you touch the sample test surface or the sample.

---



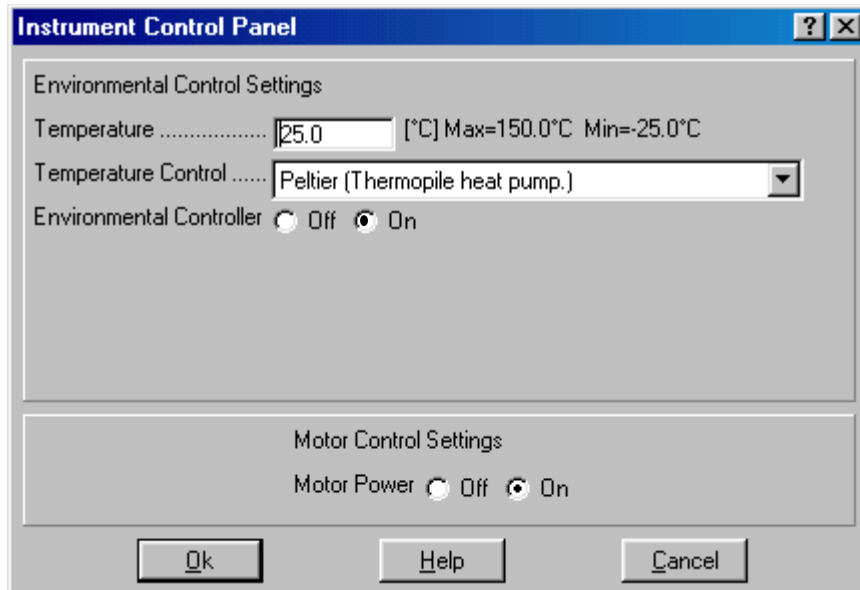


Figure 2-33. Instrument Control Panel Showing Peltier Control Options

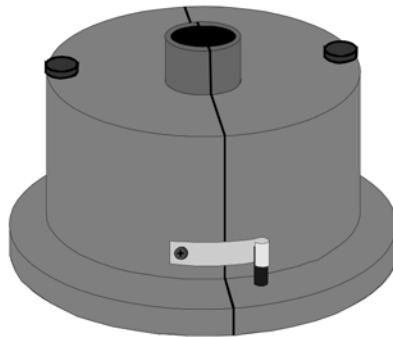


Figure 2-34. Humidity Cover

# Fluid Bath

## Description

The Fluid Bath offers precise control of sample temperature using an open fluid re-circulant system. The lower test tool is mounted within the Bath Well, around which flows thermally controlled fluid supplied by a circulator. The temperature of the lower tool is measured by the bath PRT, which mounts through the Bath Well into the Motor. You can choose to control the temperature of either the lower tool or circulator fluid.

The Circulator regulates the temperature of the bath fluid, and pumps the fluid through the Fluid Bath. The circulator (as supplied by TA Instruments), is connected to, and is under the control of, the test station and software. The fluid circulated through the bath is maintained at the temperature selected in Orchestrator. The circulator has its own fluid temperature regulation, which can optionally be used as the temperature control loop for the Bath.

## Installation of Fluid Bath

The fluid bath is mounted onto the motor using a threaded collar.

Refer to Figure 2-35 while performing the following steps to install the bath:

1. Raise the stage to maximum height and remove the upper test tool.
2. Slide the Oven all the way to the left.
3. Turn off the Motor.
4. Position the motor anvil with the knob facing the front of the instrument.
5. Position the bath with the access port facing the front of the instrument, and the alignment pin, which is located on the inside diameter of the bath housing (Figure 2-23), aligned with the corresponding notch (which should be located toward the rear of the instrument) machined into the motor housing.
6. Mount the bath onto the motor by placing the threaded collar onto the threads machined into the motor and seating the alignment pin into the notch machined into the motor housing. It may be necessary to rotate the bath back and forth until the pin falls into the notch.
7. Tighten the threaded collar then tighten the knob on the motor anvil (hand tighten only - do not over-torque).
8. Install the lower tool PRT as follows:
  - a. Ensure that the proper PRT is selected from the three lower tool PRTs available for use with the ARES Bath:

For:	Lower Fixture PRT part number
Lower Plate (Parallel Plate or Cone and Plate Geometries)	700-02647
Lower Cup (Standard Couette Geometry)	700-02647-1
17mm Lower Cup (Shallow Couette Geometry)	700-02647-2

- b. Using very little force, place the PRT plug (keyed end) through the hole in the bath, and onto the receptacle that is installed in the motor.

- c. Gently push down on the PRT and rotate it until the PRT slips into the receptacle (the keyed end of the PRT then slips into the key slot machined into the receptacle). When properly installed, Orchestrator should indicate ambient temperature.

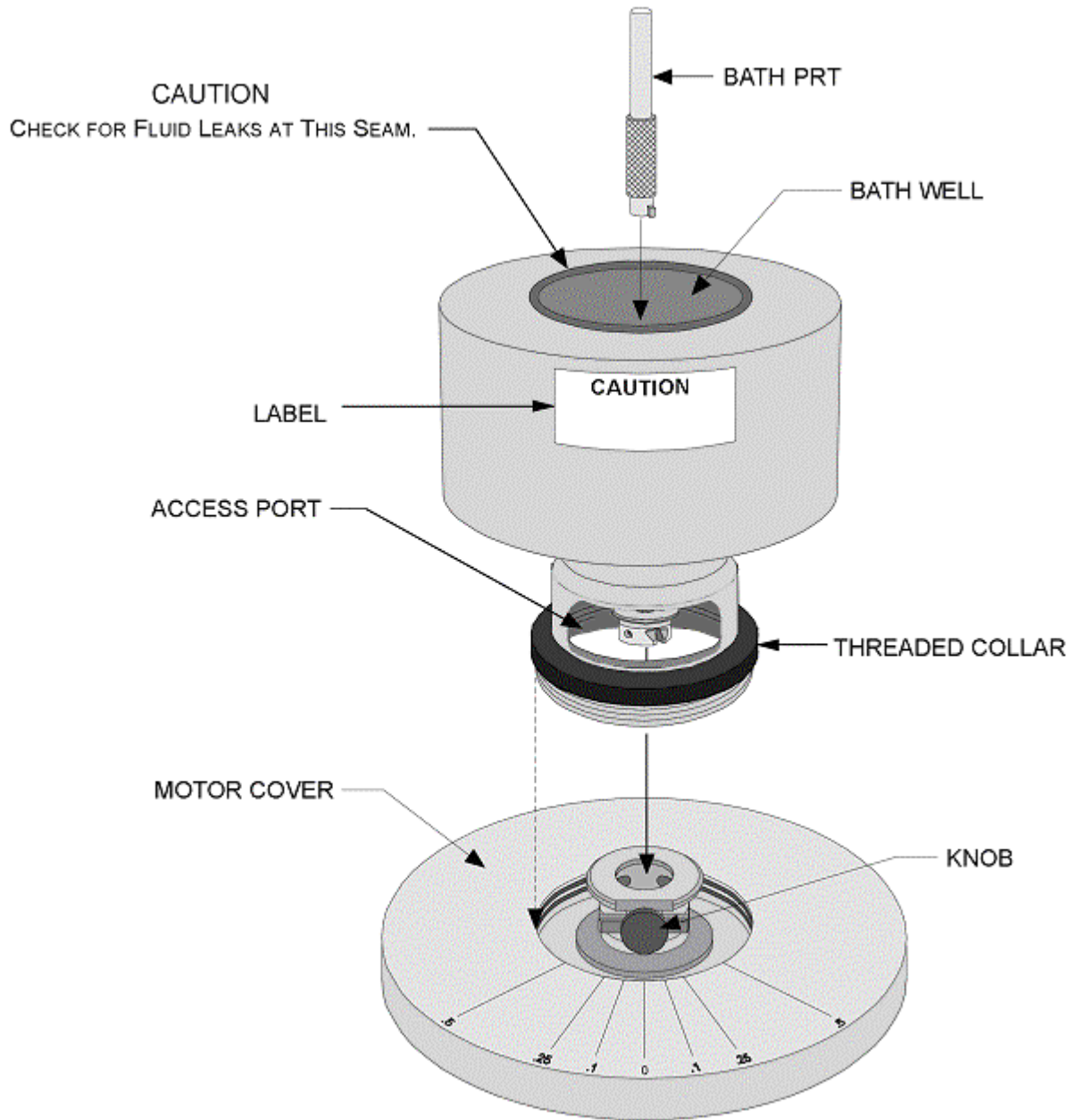


Figure 2-35. Fluid Bath Installation

## Lower Tool Installation

To install a lower tool into the fluid bath, place the tool into the bath (Figure 2-36) and apply downward pressure until the tool is seated flush against the bath.



**CAUTION:** Never place any lower tool into the bath if the temperature of the lower tool is cooler than that of the bath. Placing a tool into a warmer bath will result in expansion of the tool during use. After expansion, the tool may not be removable without damaging your bath.

We suggest that you partially insert the tool by placing a phenolic spacer between the upper lip of the lower tool, and the surface of the bath. Allow the lower tool temperature to match that of the bath, then remove the spacer and fully insert the lower tool.

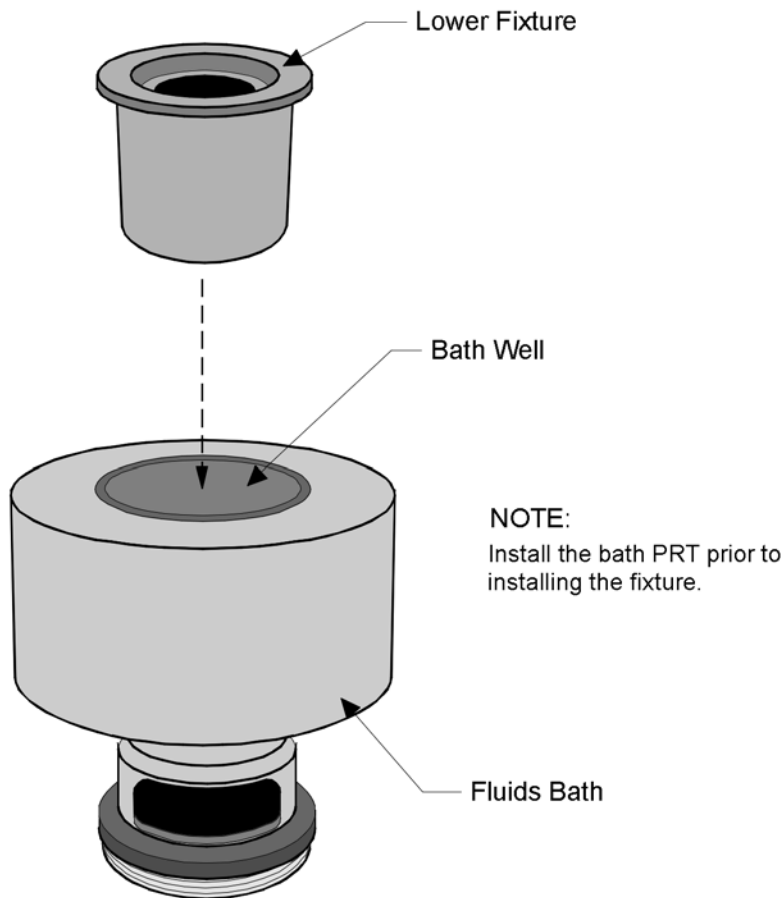


Figure 2-36. Installation of Lower tool into Fluids Bath

## Circulator Connections

Figure 2-37 shows fluid connections between the bath and Test Station. The circulator should be positioned on the floor below the work area. Make sure all hoses are installed completely onto their respective hose barbs and that the hose clamps are tight. Connect the supplied cable between the Circulator RS-232 connector, and the Test Station Signal Panel CIRCULATOR connector.

### Inlet Line Clamp

A clamp should be installed on the hose running from the circulator pump outlet to the fluid bath inlet. This clamp is critical to adjust the flow to the bath. If the clamp is not installed, or the flow not adjusted properly, it is possible to overflow the Fluid Bath and cause significant damage to the test station motor.

The clamp should be set by placing it on the inlet hose (circulator off) and completely closing the clamp as tight as it will go (finger tight). Make an index mark on the clamp knob, and then using the index for reference, open the clamp two to four complete turns. The number of turns the clamp is opened will depend upon the make and model of circulator used. We have found that when using the Julabo FS-18, (supplied with new systems) the clamp should be opened four turns. For older NesLab chillers two turns was effective. However, these values should be taken as guidelines only, and you should monitor the flow, and bath, to ensure that the flow rate to the bath is correct for their specific system.

When the clamp is set correctly you can feel (by holding the outlet hose) strong fluid flow through the bath outlet hose (it will pulsate somewhat), and the bath will stabilize at the desired temperature in about 30 minutes. If the clamp is closed too much, the flow through the bath outlet hose will feel weak or nonexistent, and the temperature will continuously oscillate without stabilizing. If the clamp is opened too much the bath will eventually overflow. The bath should be monitored, and if any fluid appears at the top seam between the bath cup and body (Figure 2-35), the clamp should be tightened immediately.

## Filling the Circulator

Depending on the type of circulator in use and the desired operating range of the circulator, fill the circulator with fluid as specified in Table 2-10. Refer to your Bath documentation for specific circulator filling and operating guidelines, as well as other bath fluid options for your application. Also refer to the bath fluid MSD for guidelines regarding the safe handling of your particular bath fluid.

Table 2-10. Circulator Operating Ranges Using Various Bath Fluids

Desired Operating Range of Circulator (bath temperature range is slightly less)	Fluid
-20°C to +30°C	100% Dow Corning® Syltherm® XLT
+1°C to +99°C	100% water
-5°C to +100°C	50% ethylene glycol / 50% water
+40°C to +110°C	100% ethylene glycol
-20°C to +150°C	Julabo® Thermal H10S

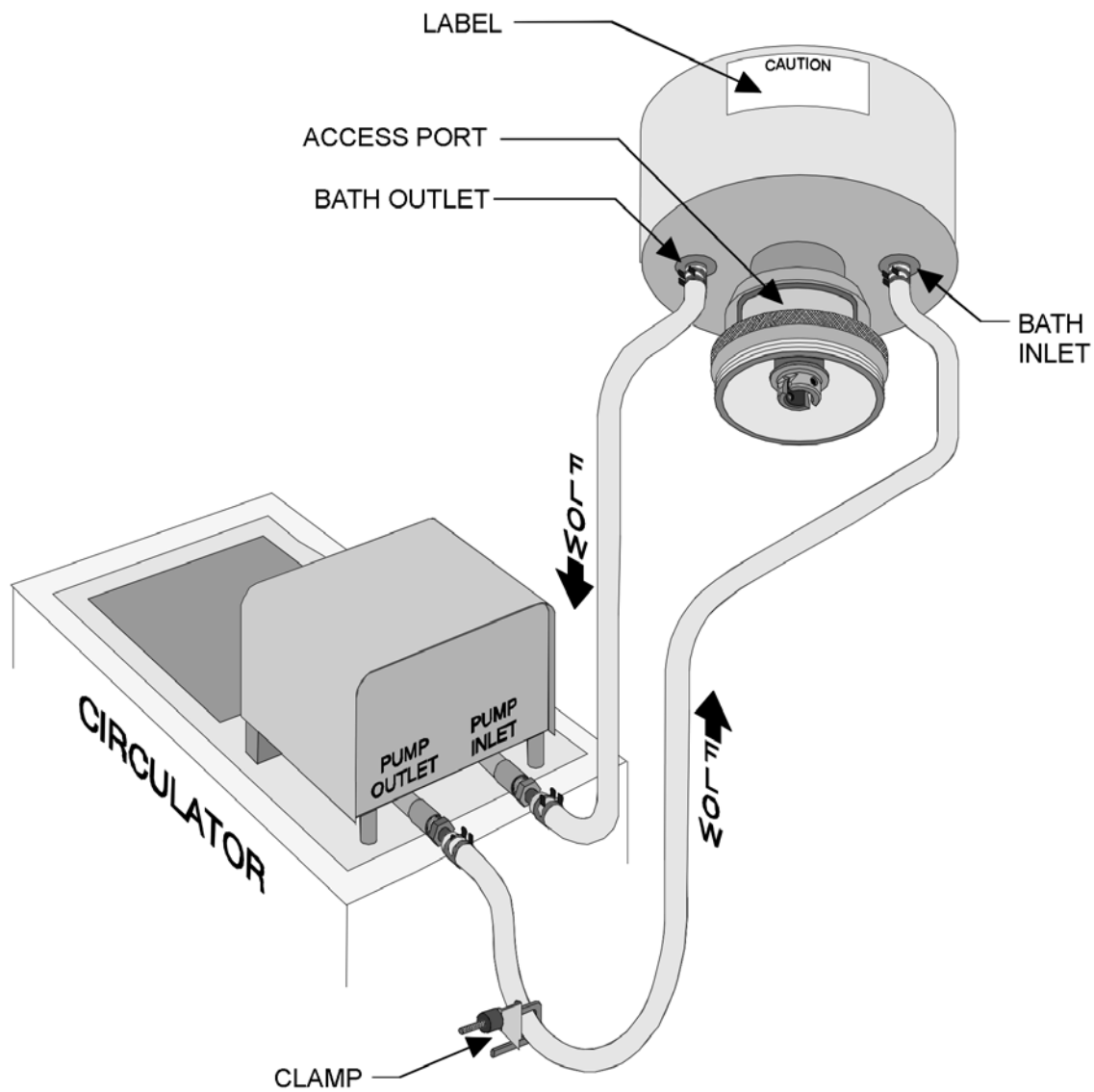


Figure 2-37. Fluid Bath: Fluid Connections

## Fluid Bath Configuration in Orchestrator™

When you command a temperature, the Orchestrator software uses the Instrument Configuration to determine the environmental system currently in use, and establish operating conditions. Prior to operating the fluid bath, access the *Instrument Configuration* function, located under the *Service* function of the Utilities menu, and set up the instrument using the guidelines shown in Figure 2-38.

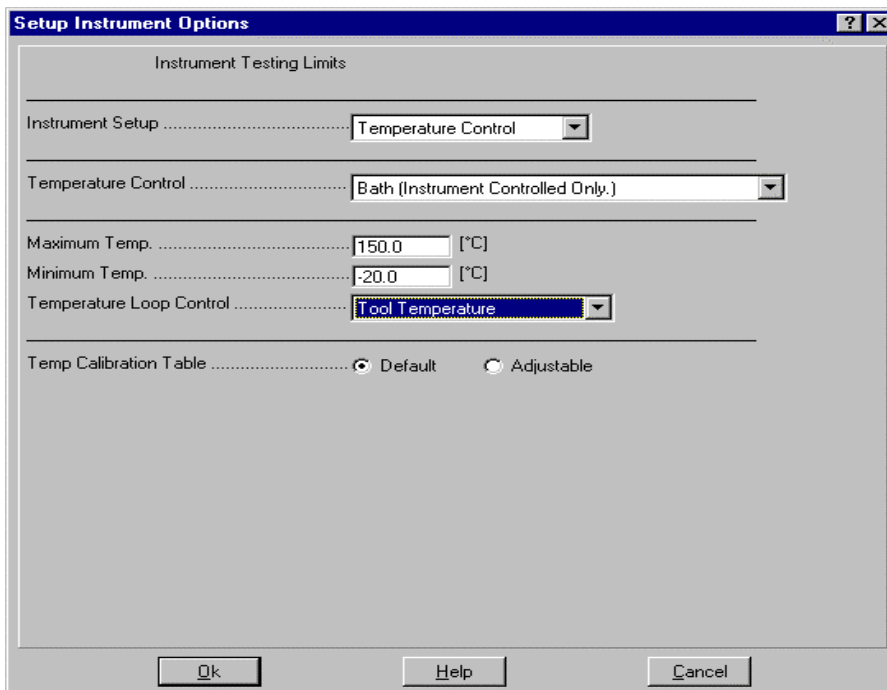


Figure 2-38. Setup Instrument Options Form

Used to Input the Fluid Bath System Configuration

### NOTES:

- (1) Make sure the following are selected
  - Instrument Setup = TEMPERATURE CONTROL
  - Temperature Control = BATH (INSTRUMENT CONTROLLED ONLY)
- (2) Ensure that the maximum and minimum temperatures corresponding to the desired circulator operating range and fluid (see the previous topic *Filling the Circulator*) are entered in the Max and Min Temperature fields.
- (3) Select either Temperature Loop Control option, using the following information as a guideline:
  - **Circulator Temperature:** When selected, the temperature of the fluid *in the circulator* is maintained at the commanded temperature. The lower tool PRT is independent of the circulator temperature, and due to its location, will report temperatures that are slightly different than the commanded (circulator) temperature. The circulator temperature control should be used when running step or ramp type temperature studies where a controlled, steady, temperature change, or speed, is more critical than actual sample temperature.

NOTE (continued):

- **Tool Temperature:** When selected, the temperature of the lower test tool is maintained at the commanded temperature. Tool Temperature control is the recommended setting for isothermal (and some step) testing, where controlling the actual sample temperature is most important. When the commanded temperature has been achieved, wait 20 to 30 minutes at the commanded temperature before starting the test to allow the bath and sample to stabilize.
- (4) For normal applications set the Temperature Calibration Table to Default. For critical temperature work, the adjustable option can be used to enter calibration "offsets" for specific temperature "setpoints". Refer to the Orchestrator online Help for details on how to set up this feature.

## PID Loop Setup

When using environmental control systems, errors between commanded and actual temperature are driven to zero by a PID (Proportional + Integral + Derivative) loop control system. The PID loop settings (Figure 2-39) affect how the bath settles at a set-point temperature. The values are affected by circulator fluid used, as well as the circulator specifications. As the bath reaches a setpoint there will typically be a few degrees of overshoot, after which the temperature will oscillate a few times and then stabilize. Typical stabilization times are 15 minutes to reach  $0.1^{\circ}\text{C}$  and 35 minutes to reach a temperature stable at  $0.01^{\circ}\text{C}$ . By modifying the Proportional Band (P coefficient) less overshoot can be achieved, but there will typically be more oscillations before the bath stabilizes, so the overall stabilization time will be longer.

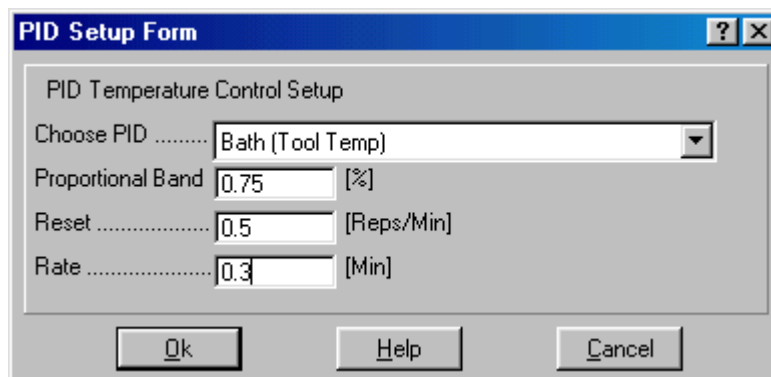


Figure 2-39. PID setup form showing Fluid Bath settings.

The values listed in Table 2-11 should be used as guidelines, and will work for most applications. However, you may need to experiment somewhat to determine their baths best PID settings for their system, or specific applications. The Orchestrator Online Help has a complete description of how to determine and tune the PID coefficients.



Table 2-11. PID Values for Various Circulators and Fluids

Circulator	Julabo® FS-18	Julabo® FS-18	NesLab V5 firmware	NesLab V4 (or below) firmware
Bath Fluid	100% Water	50% Water 50% Ethylene Glycol	100 % Water	100% Water
Proportional Band	0.75	0.75	1.00	3.00
Reset	0.50	0.25	0.75	0.75
Rate	0.30	0.67	0.25	0.25

## Fluid Bath Operating Requirements

The Fluid Bath can operate only if the following conditions are met:

- The Fluid Bath is selected as the current environmental system.
- The circulator must be filled, on, and circulating fluid through the bath.
- The circulator must be connected to the test station via the correct RS-232 cable.
- The clamp is set properly.

## Fluid Bath Operation

The Fluid Bath is operated using the Instrument Control Panel (Figure 2-40). The desired temperature is set in the Temperature input field. When testing at higher than ambient temperature, the circulator set-point must always be set to a temperature greater than the desired bath temperature.

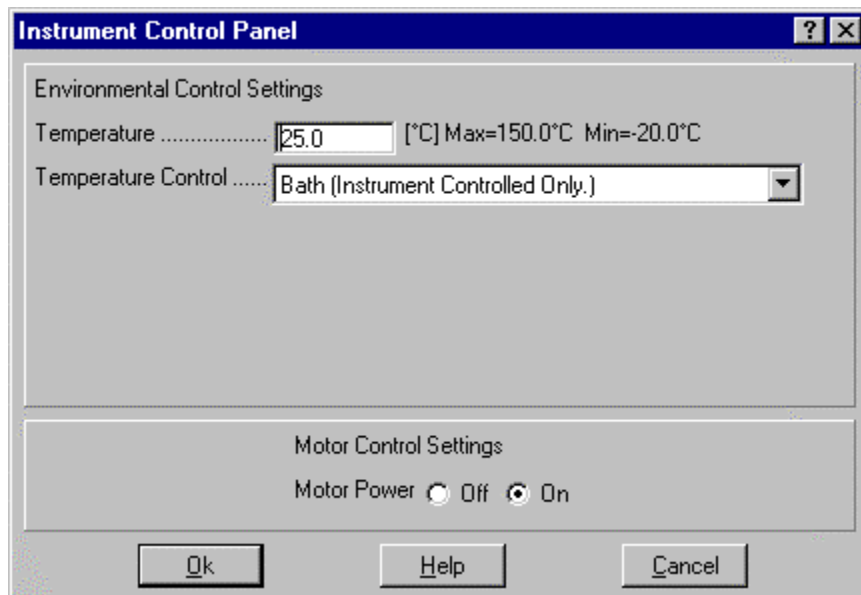


Figure 2-40. Instrument Control Panel Showing Fluid Bath Control Options



---

---

# Chapter 3

## Test Descriptions

### Introduction

#### *Stress, Strain, and Viscoelasticity*

#### Definition of Terms

Elasticity is the ability of a material to store deformational energy, and can be viewed as the capacity of a material to regain its original shape after being deformed. Viscosity is a measure of the ability of a material to resist flow, and reflects the ability of the material to dissipate deformational energy through flow. Material will respond to an applied force by exhibiting either elastic or viscous behavior, or more commonly, a combination of both mechanisms. The combined behavior is termed viscoelasticity.

In rheological measurements, the deformational force is expressed as the stress, or force per unit area. The degree of deformation applied to a material is called the strain. Strain may also be expressed as sample displacement (after deformation) relative to pre-deformation sample dimensions. Sample deformations can be in the form of either simple shear (where the material is deformed in a plane while confined between two surfaces), or linear deformations (where the material is either compressed or extended).

#### Hooke's and Newton's Laws

Hooke's law describes the mechanical behavior of an ideal solid. Hooke's law for shear deformations states that if a shear strain  $\tau$  is applied to an ideal solid, a shear stress  $\gamma$  develops in the material in direct proportion to the strain:

$$\tau = G\gamma$$

The proportionality constant in equations for shear ( $G$ ) is known as the modulus of the material. The modulus of a material is a measure of its stiffness, or ability to resist deformation. Linear stress-strain behavior is characterized by the modulus remaining unchanged as strain is varied. The region where the modulus remains constant as strain is changed is called the linear region. The linear region is also called the Hookean region of the material.

Newton's law describes the mechanical behavior of an ideal viscous fluid. When a fluid moves by virtue of being pushed through a pipe, or dragged through a screw in an extruder, etc., the movement is termed shear. Newton's law relates the shear stress ( $\tau$ ) to the *rate* of strain (or shear rate)  $d\gamma/dt$ :

$$\tau = \eta \frac{d\gamma}{dt} \quad \text{where: } \eta = \text{coefficient of viscosity}$$

A fluid is said to be Newtonian if the viscosity does not depend upon the strain rate. An analogous equation can be written for tensile testing, where the tensile stress ( $\sigma$ ) is related to the tensile strain ( $\epsilon$ ) by:

$$\sigma = \mu \frac{d\varepsilon}{dt}$$

In a non-Newtonian fluid, the viscosity is not constant, but is a function of strain rate. Many polymer solutions are non-Newtonian in behavior because their viscosity decreases as shear rate is increased. This is called shear thinning or pseudoplasticity. In the opposite effect, shear thickening or dilatancy, the viscosity increases with increasing shear rate. This is seen in some concentrated aqueous dispersions of clays and sands.

## Steady Shear Testing

Steady Shear Testing uses continuous rotation to apply strain. When a steady shear rate is reached, the shear stress ( $\tau$ ) is measured as a function of the shear rate ( $d\gamma/dt$ ). The stress-to-shear-rate ratio yields the steady shear viscosity ( $\eta$ ). Measurements are typically made over a wide range of shear rates to study the shear rate dependence of the sample.

## Dynamic Mechanical Testing

### Definition of Variables

Dynamic mechanical testing involves the application of an oscillatory strain to a sample. The resulting sinusoidal stress is measured and correlated against the input strain, and the viscous and elastic properties of the sample are simultaneously measured. If the sample behaves as an ideal elastic solid, then the resulting stress is proportional to the strain amplitude (Hooke's Law), and the stress and strain signals are in phase. If the sample behaves as an ideal fluid, then the stress is proportional to the strain rate, or the first derivative of the strain (Newton's Law). In this case, the stress signal is out of phase with the strain, leading it by  $90^\circ$ .

For viscoelastic materials, the phase angle shift ( $\delta$ ) between stress and strain occurs somewhere between the elastic and viscous extremes. The stress signal generated by a viscoelastic material can be separated into two components: an elastic stress ( $\tau'$ ) that is in phase with strain, and a viscous stress ( $\tau''$ ) that is in phase with the strain rate ( $d\gamma/dt$ ) but  $90^\circ$  out of phase with strain. The elastic and viscous stresses are sometimes referred to as the in-phase and out-of-phase stresses, respectively.

The elastic stress is a measure of the degree to which the material behaves as an elastic solid. The viscous stress is a measure of the degree to which the material behaves as an ideal fluid. By separating the stress into these components, both strain amplitude and strain rate dependence of a material can be simultaneously measured.

The viscous and elastic stresses can be related to material properties through the ratio of stress to strain, or modulus. Thus, the ratio of the elastic stress to strain is referred to as the elastic (or storage) modulus ( $G'$ ), which represents the ability of a material to store energy elastically. The ratio of viscous stress to strain is referred to as the viscous (or loss) modulus ( $G''$ ), and is the measure of a material's ability to dissipate energy. The complex modulus ( $G^*$ ) is a measure of the overall resistance of a material to deformation. If these measurements are made using a linear geometry instead of a shear geometry then the letter E is used to represent the modulus, instead of G.

In some cases it is useful to define the ratio of sample strain to sample stress. This is known as a compliance, and is represented by a J in shear testing, and a D in linear testing. As in case of modulus values it is possible to define both elastic (J' or D') and viscous (J'' or D'') components to the complex compliance (J\* or D\*).

The ratio of the viscous modulus to the elastic modulus is the tangent of the phase angle shift between stress and strain, or  $\tan \delta$ .  $\tan \delta$  is a measure of the damping property of the material.

Table 3-1 and Table 3-2 summarize the variables frequently used in dynamic mechanical testing for both linear and shear testing geometries.

Table 3-1. Dynamic Mechanical Variables -- Shear Geometries

Symbol	Variable	Equation
$\tau^*$	Complex Stress Amplitude	
$\gamma^*$	Complex Strain Amplitude	
$\delta$	Phase Shift	
$G^*$	Complex Modulus	$\tau^*/\gamma^*$
$G'$	Elastic Modulus Storage Modulus	$G^* \cos(\delta)$
$G''$	Viscous Modulus Loss Modulus	$G^* \sin(\delta)$
$J^*$	Complex Compliance	$\gamma^*/\tau^*$
$J'$	Elastic Compliance Storage Compliance	$J^* \cos(\Delta)$
$J''$	Viscous Compliance Loss Compliance	$J^* \sin(\Delta)$
$\eta^*$	Complex Viscosity	$G^*/\omega$
$\eta'$	In phase Viscosity	$\eta^* \sin(\delta)$
$\eta''$	Out of phase Viscosity	$\eta^* \cos(\delta)$

Table 3-2. Dynamic Mechanical Variables -- Linear Geometries.

Symbol	Variable	Equation
$\sigma^*$	Complex Stress Amplitude	
$\epsilon^*$	Complex Strain Amplitude	
$\delta$	Phase Shift	
$E^*$	Complex Modulus	$\sigma^*/\epsilon^*$
$E'$	Elastic Modulus Storage Modulus	$E^* \cos(\delta)$
$E''$	Viscous Modulus Loss Modulus	$E^* \sin(\delta)$
$D^*$	Complex Compliance	$\epsilon^*/\sigma^*$
$D'$	Elastic Compliance Storage Compliance	$D^* \cos(\delta)$
$D''$	Viscous Compliance Loss Compliance	$D^* \sin(\delta)$
$\mu^*$	Complex Viscosity	$E^*/\omega$
$\mu'$	In phase Viscosity	$\mu^* \sin(\delta)$
$\mu''$	Out of phase Viscosity	$\mu^* \cos(\delta)$

## Measurement Method

During dynamic mechanical testing, the ARES control computer makes a digital cross-correlation of measured strain and force by comparing the amplitude and phase shift between the imposed motion (strain) and the force (stress). When a test is started, the computer measures strain and force 2,048 times to determine the average amplitude and phase shift of both. The measurements are made relative to two reference sine waves (command) of fixed amplitude, and having phase angles of 0° and 90°, as shown in Figure 3-1A.

The result is a strain and force phaser relative to the reference as shown in Figure 3-1B.

Using fundamental geometric techniques, the phasers in Figure 3-1B can be rotated so that strain becomes the reference axis as shown in Figure 3-1C. The force vector can now be defined in terms of an in-phase and out-of-phase component of force proportional to angle  $\theta$ .

Using equations appropriate to the geometry under test, the average force phaser is converted to stress and the average angular strain phaser is converted to percent strain. Dividing the stress by the strain produces the complex modulus,  $G^*$ , which indicates the total energy required to deform the material. Multiplying  $G^*$  by the cosine of the phase angle gives the in-phase component of the stress,  $G'$ , which is proportional to the energy stored elastically. Multiplying  $G^*$  by the sine of the phase angle gives the out-of-phase component of the stress,  $G''$ , which is proportional to the amount of energy lost to viscous dissipation.

## Data Correlation

All dynamic mechanical data correlation is performed on between 1 and 64 cycles of oscillation, depending upon the testing frequency, as shown in Table 3-3.

Table 3-3. Single Point Measurement Data Correlation

Frequency (rad/sec)	Cycles of Correlation
< 2	1
<4	2
<8	4
<16	8
<100	16
<500	64

The control computer samples 2,048 data points *regardless of the test frequency*. The number of cycles of correlation determines the number of data points sampled per cycle. For example, at frequencies less than 2 rad/sec, the computer samples all 2,048 data points during one cycle of strain and force. At 500 rad/sec, only 32 data points are sampled. At higher frequencies fewer points per cycle are used, however the data is collected over multiple cycles, so the actual number of data samples still totals 2048.

At least one complete cycle is required for correlation, resulting in measurement time being inversely proportional to frequency. This is an important consideration when observing materials that change rapidly over time, such as cures involving gels and thermosets where sample material properties may differ dramatically between test start and completion.

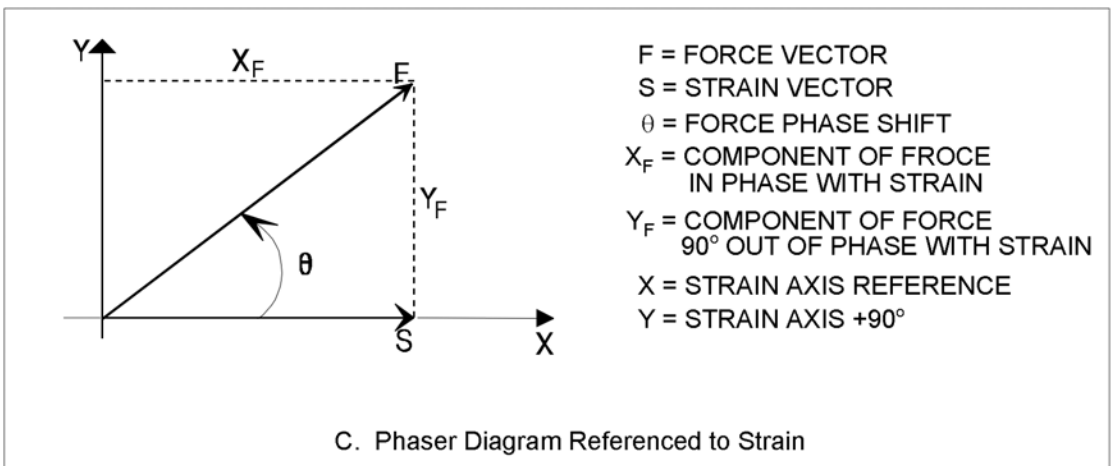
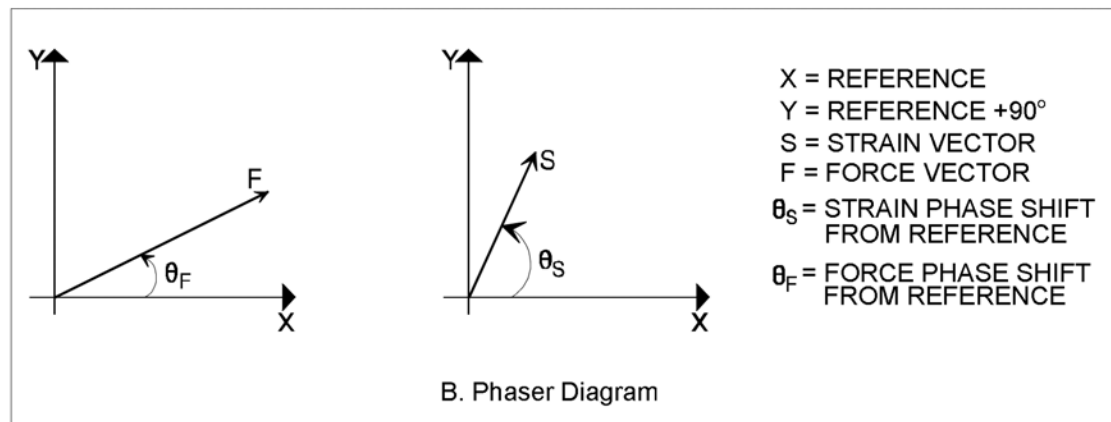
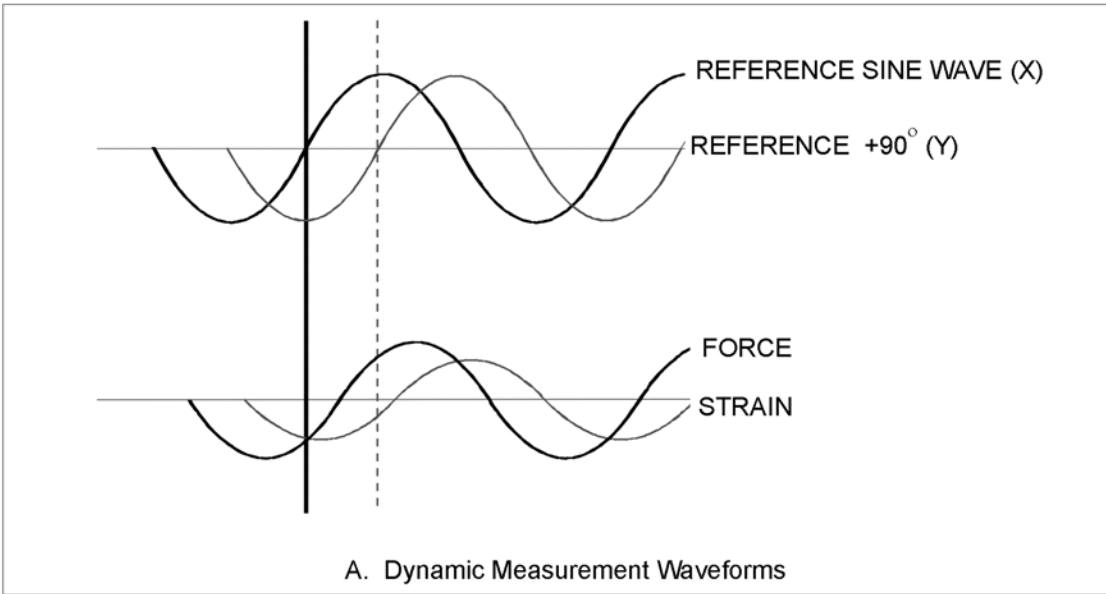


Figure 3-1. Dynamic Waveforms and Vectors



## *Documents Describing Instrument Software Operation*

The interface between you and the instrument is the TA Orchestrator software, the latest generation of instrument operating software. However, menu and function names may change without notice during subsequent software releases. Please refer to the Orchestrator Online Help system for specific details regarding the software version you are currently running.

Comprehensive procedures concerning Orchestrator operation are provided in the two TA Instruments documents:

- *Orchestrator™ Getting Started Guide*. This document contains information on the operation of the system software.
- Orchestrator™ Online Help system.

Please refer to the preceding documents (as well as this manual) prior to, and during test setup and execution.

# Test Modes

The ARES is capable of performing dynamic, steady and transient (time-based) mechanical tests. These are organized in Orchestrator under the following categories:

- Strain Controlled - Dynamic -- Dynamic (oscillatory) mechanical measurements where strain is controlled and stress is measured.
- Strain Controlled - Steady -- Steady mechanical measurements where strain is controlled and stress is measured. After the sample is allowed to reach "steady state", measurements are averaged over a period of time.
- Strain Controlled - Transient -- Static time based tests where strain is controlled and stress is measured. Data are collected rapidly, to look at the sample response over time to an applied deformation.
- Stress Controlled - Transient -- Similar to Strain Controlled Transient tests, but where stress is controlled and strain is measured.

Specific categories and tests are selected from within the *Edit/Start Test* function of Orchestrator. The available tests used with the ARES are:

## Strain Controlled – Dynamic

- Dynamic Single Point Measurement
- Frequency Sweep
- Dynamic Temperature Step
- Frequency/Temperature Sweep
- Dynamic Strain Sweep
- Dynamic Time Sweep
- Dynamic Temperature Ramp
- Multiwave Single Point
- Multiwave Temperature Ramp

## Strain Controlled – Steady

- Steady Single Point
- Steady Rate Sweep

## Strain Controlled - Transient

- Stress Relaxation
- Arbitrary Waveshape
- Step Shear Rate
- Thixotropic Loop
- Force Gap Test
- Torque/Normal Relaxation
- Multiple Extension Mode
  - Constant Force mode (Creep/Recovery)
  - Strain Rate mode
  - Hencky Strain Rate mode
  - Rim Shear (Squeeze Flow) mode
- Steady Step Rate Temperature Ramp

**Stress Controlled Transient**

- Constant Stress Test
- Stress Ramp Test

# Strain Controlled Dynamic Test Methods

## *Dynamic Single Point Measurement*

### Functional Description

The Single Point Measurement test makes a single measurement at a fixed frequency, strain and temperature.

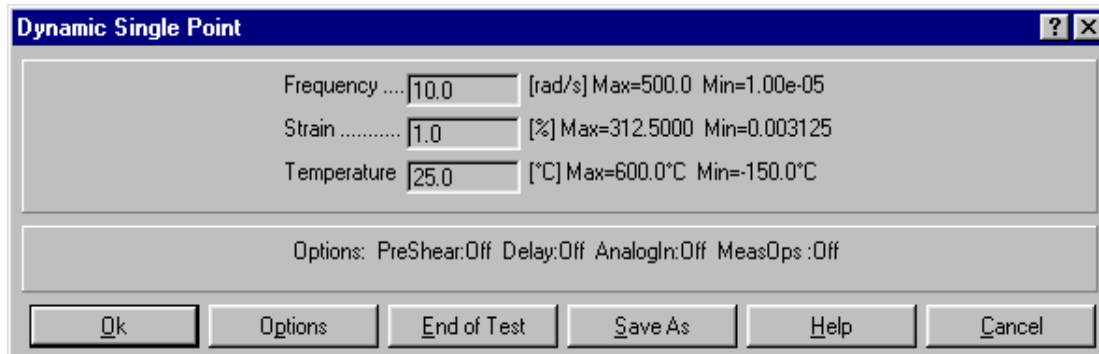


Figure 3-2. Dynamic Single Point Test Set-Up Screen

### Suggested Uses

Suggested uses for single point measurement are as follows:

- Determination of unknown range response limitations of a new sample material
- Determination of parameters for the design of new tests
- Determination of force generated at various conditions and temperatures for the purpose of keeping force within the range of the transducer during sweeps.

### Test Options

The following test options can be selected for use with the single point measurement (see section on Testing Options for further details):

- Steady PreShear
- Delay Before Test
- Analog Data Input
- Correlation (Measurement) Options

# Dynamic Time Sweep

## Functional Description

The Time Sweep test takes successive measurements at constant temperature, frequency, and strain at a selected interval.

The time required to make a measurement is frequency dependent. At frequencies less than 2.0 rad/sec (0.3 Hz) it is equal to the time required to complete approximately 1.5 cycles of oscillation. At high frequencies the limit is approximately 2 second per measurement. Adjusting the Correlation Delay (see "Measurement Options") can also have an effect on the time required to make a measurement.

If a "Time Per Measure" is entered that is less than the amount of time required to complete the measurement (Figure 3-3), the control computer will take data points at its maximum speed, in accordance with frequency. If many points are to be taken over a short time period, a short time/measurement, such as 2.0 sec, could be used. This sets the instrument to take measurements as fast as possible.

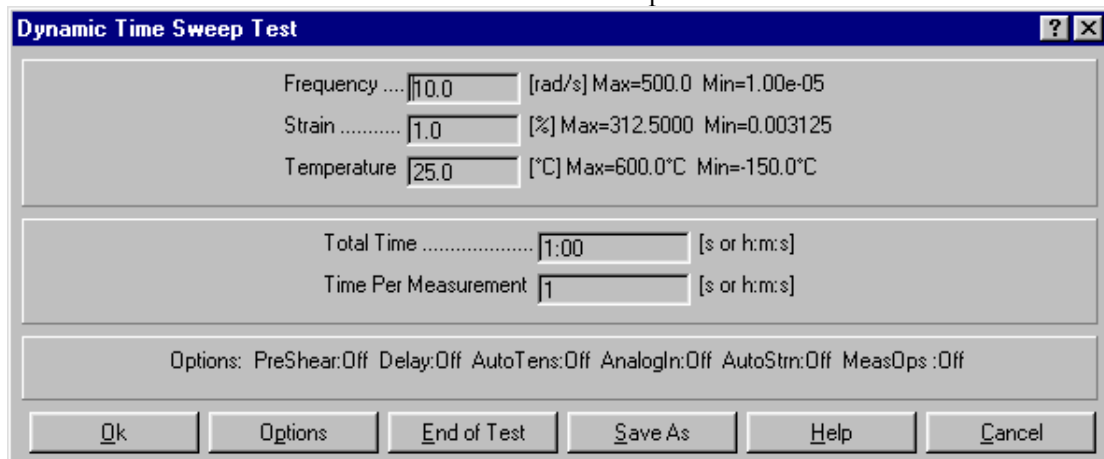


Figure 3-3. Dynamic Time Sweep Test Set-up Screen

## Suggested Use

Time sweep provides a means of monitoring the time-dependent behavior of a material, for example thermal degradation at temperature, curing in thermosetting systems, or the build-up or breakdown of network structure.

## Test Options

The following test options can be selected for use with the time sweep:

- Steady PreShear
- Delay Before Test
- AutoTension
- Analog Data Input
- AutoStrain
- Measurements Options
  - Delay Settings
  - Strain Amplitude Control

# Frequency Sweep

## Functional Description

The Frequency Sweep test takes successive measurements at selectable frequencies while holding a constant strain and temperature. Ideally, the selected strain should be within the linear viscoelastic region of the sample. The time it takes to complete the test is highly dependent on the low end frequency selected. For example, a frequency sweep from 0.1 to 100 rad/sec (taking 10 data points per decade) will take approximately 12 minutes. Changing the range to 0.01 to 100 rad/sec increases the test time to around 2 hours, and running the test from 0.001 to 100 rad/sec may take over 20 hours.

Three types of frequency sweeps can be executed (Figure 3-4), as follows:

### Logarithmic Frequency Sweep

The logarithmic frequency sweep uses the entered frequency values as the upper and lower frequency limits of the sweep. Logarithmic frequency sweeps can be run in ascending or descending order. They can begin or end at any frequency from  $1 \times 10^{-6}$  to 80 Hz with a maximum of 500 data points measured during each decade of frequency.

Frequencies are selected by specifying initial and final frequencies, and the number of data points to measure between each decade of frequency (points per decade). The number of points per decade includes the initial frequency, but excludes the final frequency. As an example, consider a sweep conducted over a single decade of frequency: between 10 and 100 radians per second. Selecting five data points to be measured per decade divides the difference of the endpoint logarithms into five equally-spaced fractional exponents, so that six discrete frequencies are generated:

- Initial Frequency = 10 rad/sec
- Decade Frequencies = 15.9, 25.1, 39.8, 63.1 rad/sec
- Final Frequency = 100 rad/sec

One data point is measured at each of the frequencies.

### Linear Frequency Sweep

The linear frequency sweep uses the entered frequency limits. The test starts at the initial frequency and continues until the final frequency is reached, adding the frequency increment to each subsequent frequency if the final frequency is greater than the initial value, or subtracting the increment if the final frequency is less than the initial value.

As an example, a linear sweep from 10 to 50 rad/sec, in increments of 10 rad/sec, generates frequencies of 10, 20, 30, 40, and 50 rad/sec. One data point is measured at each of the frequencies.

### Discrete Frequency Sweep

The discrete frequency sweep takes a measurement at each of up to ten selected frequencies. Discrete frequency sweeps can be run in any order, and can begin or end at any frequency within the range of the instrument.

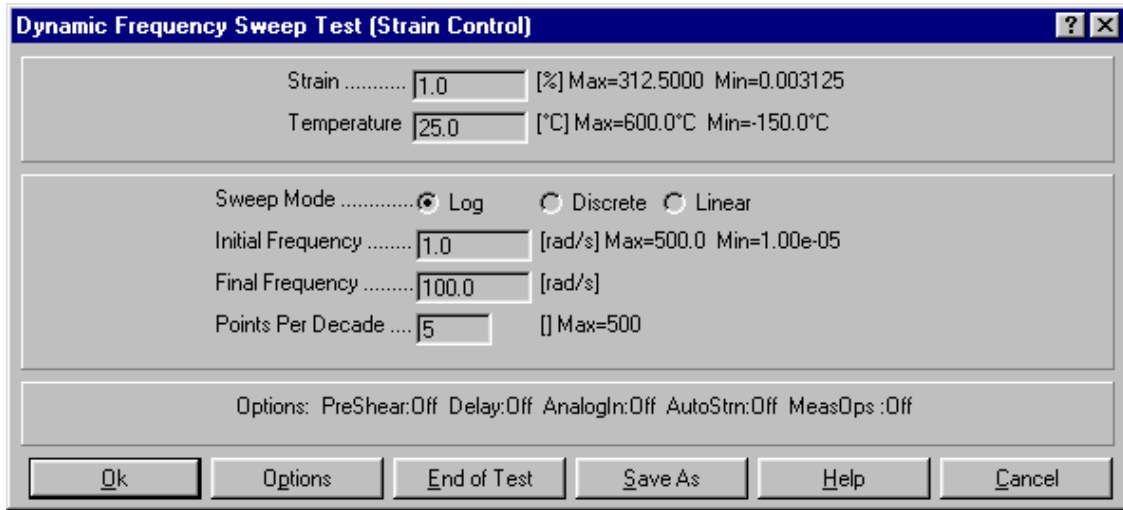


Figure 3-4. Frequency Sweep Test Set-up Screen

## Suggested Use

The frequency sweep is used to analyze frequency and time dependent behavior of samples. In general, high frequencies correspond to short time scales, and low frequencies correspond to long time scales.

## Test Options

The following test options can be selected for use with the frequency sweep:

- Steady PreShear
- Delay Before Test
- Analog Data Input
- AutoStrain Adjustment
- Correlation (Measurement) Options

# Dynamic Temperature Step

## Functional Description

The Temperature Step test takes successive measurements at selected temperatures while holding a constant frequency and strain.

Temperature is automatically incremented or decremented from selected lower and upper temperature limits by user-selected steps. A unique set of test conditions can be entered in up to four temperature ranges, or *zones*. A selected thermal "soak time" at each temperature ensures temperature equilibrium prior to measurement.

When setting up a temperature step test (Figure 3-5), you can enter a test frequency and an initial or starting temperature. Next, you can enter each individual zone final temperature, step size, soak time, and strain. Testing limits are displayed for each of these fields.

When setting strain values in this test be aware that the sample modulus can change significantly with temperature, and the torque signal generated at a given strain may vary dramatically. A strain that provides good torque and is within the linear viscoelastic region at room temperature may overload the transducer at lower temperatures. Single point tests at the temperature extremes are a good way to find the appropriate strains to set for each zone. The AutoStrain adjustment is also a good option for optimizing the torque generated in this test. Note that when AutoStrain is used it is possible to have the current strain propagate into a new zone by entering a value of zero for the strain ("Inherit" feature).

The final temperature can be any temperature within the range of the instrument, and there is no limitation on step size.

### Data Measurement Method

Each measurement is equivalent to a data point. For example, changing temperature from 10° to 100° in 10° steps produces 10 data points. Making the same temperature change in 5° steps results in 19 data points. Soak time should be chosen with consideration given to sample volume and temperature increment. Larger samples and larger step sizes require longer soak times to equilibrate. Typical soak times are from 1 to 5 minutes. The minimum soak time is zero seconds; maximum 65000 seconds.

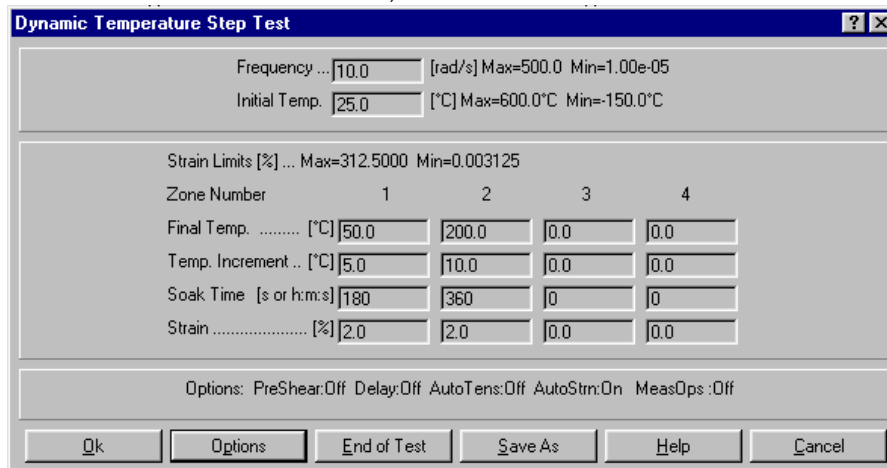


Figure 3-5. Temperature Step Test Set-Up Menu



## Suggested Use

Temperature step tests are generally used to analyze the behavior of a sample, as a function of temperature. Temperature steps are preferred over the temperature ramp method if accurate isothermal data are needed. This is because any sample thermal conductivity effects cause internal temperature gradients until the sample has had sufficient time to equilibrate at a given temperature.

## Test Options

The following test options can be selected for use with the temperature sweep:

- Steady PreShear
- Delay Before Test
- Analog Data Input
- AutoTension
- AutoStrain
- Measurement Options
  - Delay Settings
  - Strain Amplitude Control

# Dynamic Strain Sweep

## Functional Description

Strain Sweep takes successive measurements at selected step increases in strain, while holding frequency and temperature constant. Two types of strain sweeps can be executed (Figure 3-6), as follows:

### Logarithmic Strain Sweep

The logarithmic strain sweep uses the entered strain values (in dimensionless strain units) as the upper and lower strain limits of the sweep. An increment or step size, also in strain units, must be entered. Increments are performed in logarithmic steps. The number of points per decade includes the initial strain, but excludes the final strain.

As an example, consider a sweep conducted over a single decade of strain: between 10% and 100%. Selecting five data points to be measured per decade divides the difference of the endpoint logarithms into five equally-spaced fractional exponents, so that six discrete strains are generated:

- Initial Frequency = 10%
- Decade Frequencies = 15.9%, 25.1%, 39.8%, 63.1%
- Final Frequency = 100%

One data point is measured at each of the strains.

Up to 500 data points can be measured per each decade of strain. Logarithmic strain sweeps can be run in ascending or descending order, and can begin or end at any strain within the range of the instrument.

### Linear Strain Sweep

The linear strain sweep uses the entered strain limits. The test starts at the initial strain and continues until the final strain is reached, adding the strain increment to each subsequent strain if the final strain is greater than the initial value, or subtracting the increment if the final strain is less than the initial value.

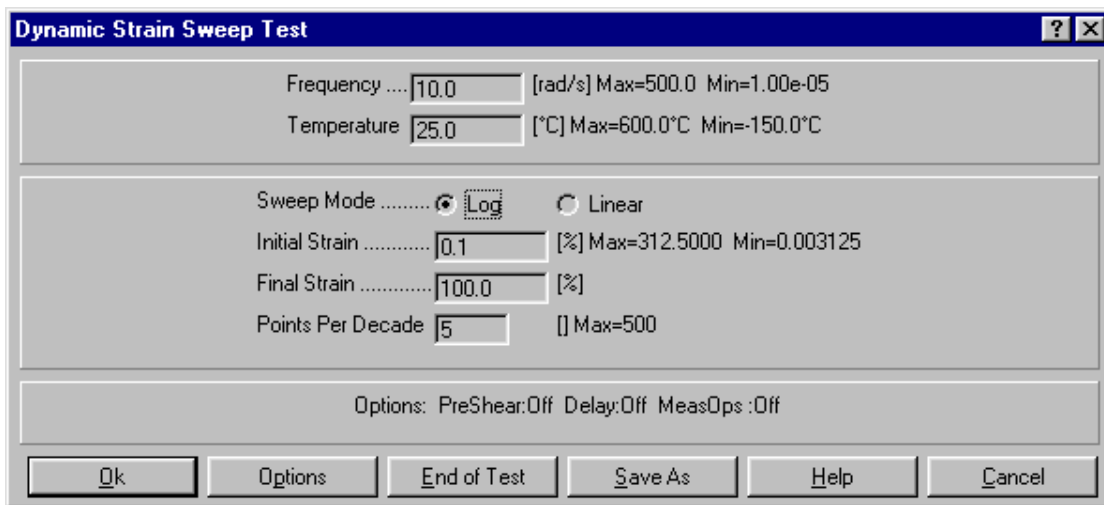


Figure 3-6. Dynamic Strain Sweep Test Set-up Screen

## Suggested Uses

Suggested uses of the strain sweep are as follows:

- Determination of the limits of linear viscoelasticity and torque levels.
- Characterization of samples that exhibit extreme nonlinear behavior, such as filled thermoplastics, thermoplastic blends, etc.

## Test Options

The following test options can be selected for use with the strain sweep:

- Steady PreShear
- Delay Before Test
- Analog Data Input
- AutoStrain
- Measurements Options
  - Delay Settings
  - Strain Amplitude Control

# Dynamic Temperature Ramp Test

## Functional Description

Temperature Ramp testing takes measurements at selectable temperature ramp rates while holding a constant frequency and strain.

Temperature is automatically incremented or decremented from selectable lower and upper temperature limits at selected rates. A unique set of test conditions can be entered in up to eight temperature ranges, or "zones". A programmable "thermal soak time" at the end of each zone ensures temperature stability prior to beginning the next ramp.

When setting up a temperature ramp test, you enter a test frequency and an initial or starting temperature. Next, you enter each individual zone final temperature, ramp rate (*i.e.*, 2 °C/min, 5°C/min, etc.), soak time, and strain.

When setting strain values in this test be aware that the sample modulus can change significantly with temperature, such that the force generated at a given strain may vary dramatically at different temperatures. A strain that provides good force and is within the linear viscoelastic region at room temperature may overload the transducer at lower temperatures. Single point tests at the temperature extremes are a good way to find the appropriate strains to set for each zone. The AutoStrain adjustment is also a good option for optimizing the force generated in this test. Note that when AutoStrain is used, it is possible to have the current strain propagate into a new zone by entering a value of zero for the strain ("Inherit" feature).

Frequency and initial temperature are entered first (Figure 3-7), and then the following parameters are entered for each zone:

### Final Temperature

Final temperature is the temperature at which the instrument stops ramping temperature while in the respective zone. The final temperature is independent of time.

### Ramp Rate

Ramp rate is the rate of change (positive or negative) that the sample material undergoes during the test. Ramp direction is set using the starting and final temperatures in a zone. The ramp rate is in units of degrees/minute. The entry field accepts any positive value for ramping rate. The actual maximum ramping rate that can be accurately maintained is a function of the size and thermal properties of the sample. If temperature control is not needed, and strain or sampling rate is the only desired change, the ramp rate should be set to 0°C/min.

### Time Per Measurement

Time Per Measurement establishes the sampling rate. When entering this value, consider the time per measurement cannot be shorter than the physical time required to make a dynamic measurement. If the time entered is less than physical time required to make a dynamic measurement the instrument will sample as fast as it can.

## Strain

Strain can be set to any value within the range of the instrument (as shown in the boundary window), but should not exceed the linear viscoelastic region of the sample material. If a value of zero is entered then this zone inherits the strain level from the previous zone. Note also that this value can be overridden if the AutoStrain function is being used.

## Soak Time After Ramp

Following the Ramp, the time period during which temperature is held at the Final Temperature before proceeding to the next zone.

Strain Limits [%] ...	Max=312.5000		Min=0.003125	
Zone Number	1	2	3	4
Final Temp. .... [°C]	200.0	0.0	0.0	0.0
Ramp Rate ..... [°C/min.]	3.0	0.0	0.0	0.0
Computed Ramp Time ..... [h:m:s]	58:20	0	0	0
Soak Time After Ramp [s or h:m:s]	5:00	0	0	0
Time Per Measure ..... [s or h:m:s]	1:00	0	0	0
Strain ..... [%]	1.0	0.0	0.0	0.0

Figure 3-7. Dynamic Temperature Ramp Test Set-Up Screen

## Suggested Uses

Suggested uses for temperature ramp tests:

- To quickly analyze the behavior of samples as a function of temperature, and to determine test parameters for subsequent Temperature Step tests.
- To simulate processing conditions that a material may experience in use by programming the relevant time/temperature profile.
- To study the response of a material to changing strain levels as a function of time and/or temperature.
- The Temperature Ramp test is one of the most flexible of all of the test modes due to its ability to handle complex time/temperature/strain profiles.

## Test Options

The following test options can be selected for use with the temperature ramp:

- Steady PreShear
- Delay Before Test
- AutoTension
- Analog Data Input
- AutoStrain
- Measurement Options
  - Delay Settings
  - Strain Amplitude Control

# MultiWave Single Point; MultiWave Temperature Ramp

## Functional Description

The MultiWave test is a dynamic test method that superimposes up to 7 harmonic frequencies on a selected fundamental frequency. This allows you to acquire data at several frequencies simultaneously, in a fraction of the time required to run a conventional frequency sweep.

MultiWave is based upon the *Boltzmann Superposition Principle*, which states that two or more simultaneous strain deformations are linearly independent of each other. Because each wave acts independently, the displacement (strain) of a point in the material is the sum of the strains caused by each wave, providing the total strain is within the linear viscoelastic region of the material. Data correlation takes place at each of the applied frequencies, using the same set of raw data, and the algorithm mathematically extracts the torque and displacement signals at the desired frequency from the total combination of signals.

In order to program this test you must provide information on the fundamental frequency:

### Frequency

The frequency of the fundamental (lowest value) used. This value must be below 2.0 rad/sec because of the way that the data correlation algorithm works.

### Strain

This is defined as the strain amplitude of the fundamental frequency.

### Temperature

This is the test temperature that will be used in your experiments.

Harmonic information is given in the next section of the test setup screen:

### Harmonic

This is an integer value by which the fundamental is multiplied by to determine the frequency for a given harmonic. A value of 0.0 (zero) is used to indicate that that zone is not used.

### Strain

This is defined as the strain amplitude of the given harmonic. Note that a value of 0.0 (zero) can be used to signal the instrument to make a measurement at the given harmonic without any applied strain at that frequency (which can be used to monitor distortions in the stress sine wave that can indicate nonlinear behavior).

The MultiWave setup menu is displayed following selection of either single point (Figure 3-8) or temperature ramp (Figure 3-9) test methods. Here, you must select a fundamental frequency between 2 rad/sec and  $1 \times 10^5$  rad/sec ( $0.318$  and  $1.59 \times 10^6$  Hz). The strain level for the fundamental frequency must also be entered.

You can select up to seven harmonics (multiples of the fundamental) and assign a strain to each harmonic. As a general rule, the sum of all strains should not exceed the linear viscoelastic region of the material. A strain sweep can be used to determine the limits of linear behavior if that information is not known. If a value of zero is entered for both the strain and harmonic, then that column is not used. If a nonzero frequency is entered, with a strain level of zero, data will still be acquired at that frequency. This provides a means for measuring the amount of harmonic stress generated by other frequencies, and can provide insight into the linearity of the measurement.

After all frequency components are specified, the implementation of single point measurement test and temperature ramp differ. The MultiWave Temperature Ramp test requires additional input to specify up to eight zones of temperature control. See the section on the Temperature Ramp Test for more information.

Once the strains and frequencies have been specified the resulting waveform must then be downloaded into the instrument. The “Wave” button is used to compute the resulting waveform, display the results graphically, and provide the option to download directly into the instruments waveform memory. Any subsequent change to the strain or frequency parameters requires recalculating and re-sending the waveform. The software keeps track of any additional changes, and when the test is started, reminds you to resend the waveform, if necessary.

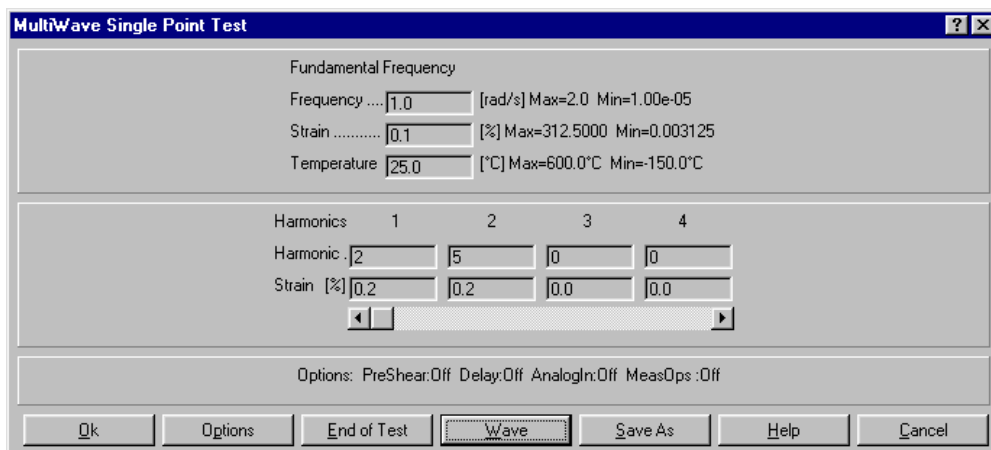


Figure 3-8. MultiWave Single Point Test Set-up Screen



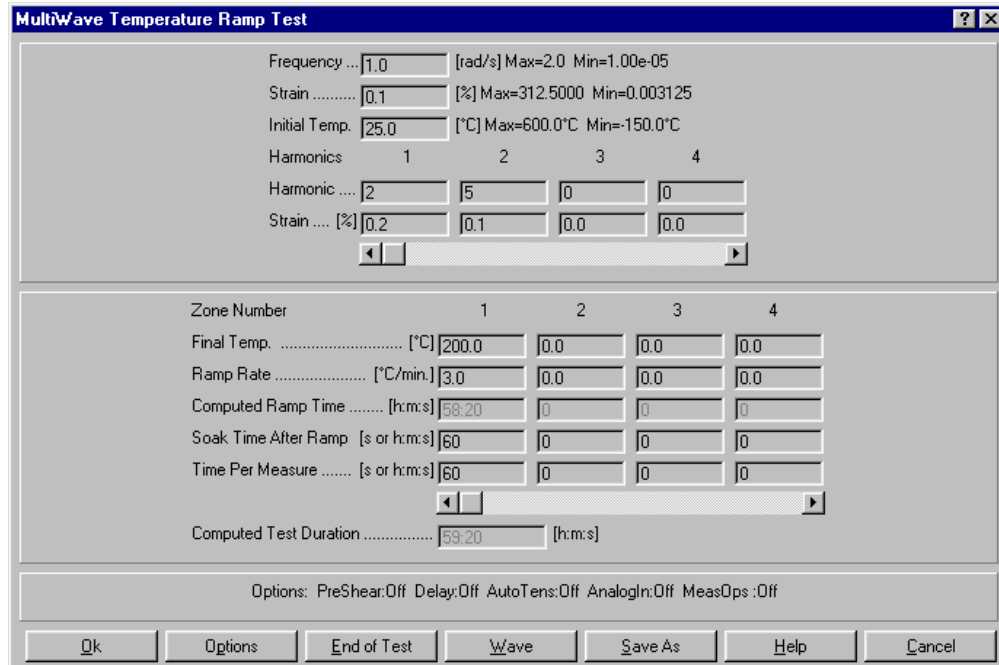


Figure 3-9. MultiWave Temperature Ramp Test Set-up Screen

## Test Options

The following test options can be selected for use with the temperature ramp:

- Steady PreShear
- Delay Before Test
- AutoTension
- Analog Data Input
- Measurement Options
  - Delay Settings
  - Strain Amplitude Control

# Frequency/Temperature Sweep

## Functional Description

The Frequency/Temperature Sweep takes successive measurements over a range of selected frequencies at a series of constant temperature "steps". At each temperature step, a frequency sweep is run (while the temperature is held constant). The temperature is then changed to the next step, allowed to equilibrate, and the frequency sweep is run again.

In a frequency/temperature sweep, entry fields for strain value and initial temperature are displayed on the host computer (Figure 3-10). You can select either a linear, logarithmic, or discrete frequency sweep then enter the frequency parameters. Thermal step size and soak time are also selectable.

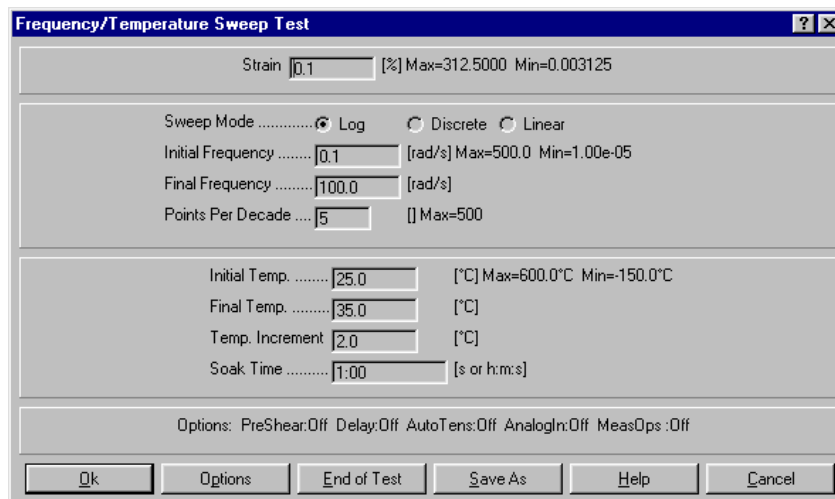


Figure 3-10. Frequency/Temperature Sweep Test Set-up Screen

## Suggested Use

Frequency/temperature sweep combines the frequency and temperature step methods to generate a group of curves which can be shifted using time-temperature superposition (TTS) along the frequency axis to extend the range of frequency characterization beyond that which is experimentally practical at the chosen reference temperature. From TTS data, a master curve can be generated for the sample.

## Test Options

The following test options can be selected for use with the frequency-temperature sweep:

- Steady Preshear
- Delay Before Test
- Analog Data Input
- AutoTension
- Measurements Options
  - Delay Settings
  - Strain Amplitude Control

# Strain-Controlled Steady Test Methods

## Steady Single Point

### Functional Description

Steady Single Point takes a single measurement while applying a steady shear deformation at a chosen shear rate. Data are taken at the commanded shear rate and temperature (Figure 3-12). Data can be collected using either of two modes:

#### Time Based

Time Based data collection takes a single measurement. Following the start of the test, the Delay Before Measure is the time period between the beginning of motor motion and the beginning of data collection. This allows time for the material to reach steady state. The Measurement Time is the period during which data are actually collected.

#### Manual Mode

Following the start of the test, Manual Mode data collection takes a single measurement when commanded to do so. Manual Mode operation is as follows:

- Start the test.
- When desired, start the measurement by clicking either the "Toggle Steady Measure" button (Figure 3-11) on the toolbar, or the *Toggle Steady Measure* function, accessed from the Control pull down menu.
- When desired, stop the measurement by clicking either the "Toggle Steady Measure" button on the toolbar, or the *Toggle Steady Measure* function.

Manual mode is designed to allow you to acquire data in the steady state region by manually monitoring the torque signal (*e.g.*, by using an external strip chart recorder), which is proportional to the sample stress. For accurate steady state data, measurements should be made at a point where all of the transients in the torque signal have disappeared, and the torque value is relatively constant.



Figure 3-11. Toggle Steady Measure Button

### Direction

For positive Rate values, Direction specifies the rotational direction of the actuator at the first commanded shear rate. Selecting Directions per Measurements of "One" results in data collection while the actuator rotates in the specified Direction. Selecting Directions Per Measurement of "Two" results in the following sequence of events:

1. Data are collected while the actuator rotates in the specified Direction
2. Data are collected while the actuator rotates in the opposite direction.
3. The data are averaged to supply the single data point that is reported by Orchestrator.

Using two directions per measurement can give more accurate results at low rates.

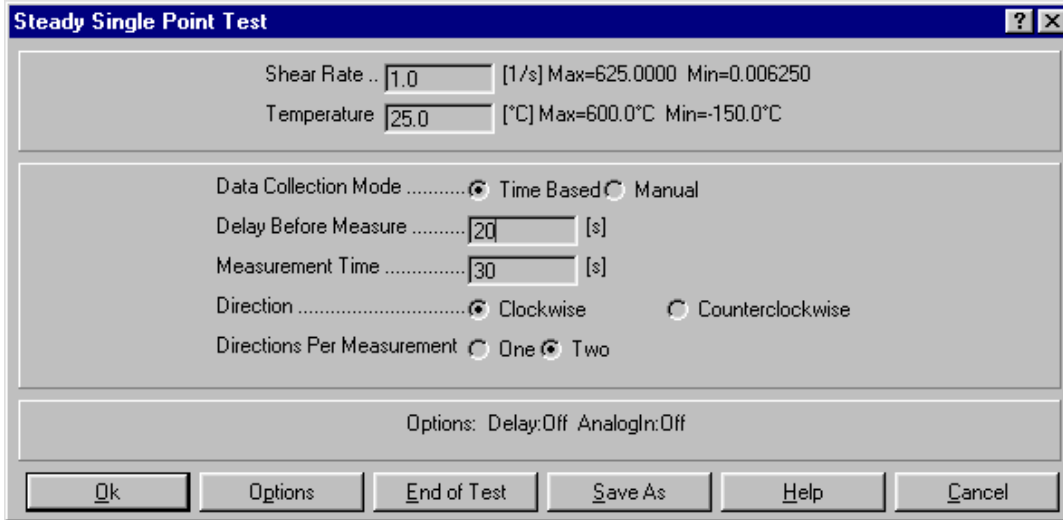


Figure 3-12. Steady Single Point Test Set-Up Screen

## Suggested Uses

Steady Single Point can be used to determine the following:

- Unknown range response limitations of a new sample material
- Parameters for the design of new tests
- Time required for a sample material to reach steady state at a given shear rate (when torque/stress signal is manually monitored, e.g. with an external strip-chart recorder)

## Options

The following test options are available for use with Steady Single Point:

- Delay Before Test
- Analog Data Input

# Steady Rate Sweep

## Functional Description

Steady Rate Sweep applies varying magnitudes of steady shear deformation, the magnitude of each deformation depending on selected shear rates (Figure 3-13). Shear rates can be generated as follows:

### Logarithmic

The logarithmic rate sweep commands rates that are logarithmically incremented, resulting in equally spaced data points when plotted as a function of logarithmically-scaled shear rate, in reciprocal seconds (1/s). Shear rates are selected by specifying initial and final rates, and the number of data points to measure between each decade of rate.

As an example, consider a sweep conducted over a single decade of rate: from 10 to 100 reciprocal seconds (1/s). Selecting five data points to be measured per decade divides the difference of the endpoint logarithms into five equally spaced fractional exponents:

Six discrete rates are generated, in succession, by taking the antilogarithm of each exponent:

- Initial Rate = 10 1/sec
- Decade Rates = 15.9, 25.1, 39.8, 63.1 1/sec
- Final Rate = 100 1/sec

One data point is measured at each of the rates.

### Discrete

The discrete rate sweep generates up to five shear rates in succession. Each shear rate is entered into a Zone.

## Data Collection Mode

Data can be collected in either time-based mode (one measurement is taken at each rate) or manual mode (one measurement is taken at selected rate).

### Time Based

Following the start of the test, Time Based data collection takes one measurement at each rate. Setting the Sweep Mode to Log commands logarithmically incremented shear rates. A Discrete Sweep Mode commands up to five unique shear rates in succession. At each shear rate, Measurement Time is the period during which data are collected. At each shear rate, Delay Before Measure is the time period between command of the current rate and the beginning of data collection.

## Manual Mode

Following the start of the test, Manual Mode data collection takes a single measurement when commanded to do so. Manual Mode operation is as follows:

- Start the test.
- When desired, start the measurement by clicking either the "Toggle Steady Measure" button (Figure 3-11) on the toolbar or the *Toggle Steady Measure* function, accessed from the Control pull down menu.
- When desired, stop the measurement by clicking either the "Toggle Steady Measure" button on the toolbar or the *Toggle Steady Measure* function.

## Direction

For positive Rate values, Direction specifies the rotational direction of the actuator at the first commanded shear rate. Selecting Directions per Measurements of "One" results in data collection while the actuator rotates in the specified direction. Selecting Directions Per Measurement of "Two" results in the following sequence of events:

1. Data are collected while the actuator rotates in the specified direction
2. Data are collected while the actuator rotates in the opposite direction.
3. The data are averaged to supply the single data point that is reported by Orchestrator.

Using two directions per measurement is necessary if the transducer changes range during the test.

The screenshot shows the 'Steady Rate Sweep Test' dialog box. The 'Temperature' field is set to 25.0 [°C] with a range of Max=600.0°C and Min=-150.0°C. The 'Sweep Mode' has 'Log' selected. 'Initial Rate' is 1.0 [1/s] (Max=625.0000, Min=0.006250) and 'Final Rate' is 100.0 [1/s]. 'Points Per Decade' is 5 (Max=500). 'Data Collection Mode' has 'Time Based' selected. 'Delay Before Measure' is 20 [s] and 'Measurement Time' is 30 [s]. 'Direction' has 'Clockwise' selected. 'Directions Per Measurement' has 'Two' selected. At the bottom, there are buttons for 'Ok', 'Options', 'End of Test', 'Save As', 'Help', and 'Cancel'. A status bar at the bottom indicates 'Options: Delay:Off AnalogIn:Off'.

Figure 3-13. Steady Rate Sweep Test set-Up Screen

## Suggested Uses

This test is used to generate "flow curves" for samples, by measuring the stress and viscosity as a function of shear rate. This can be used to characterize the non-Newtonian behavior of materials.

## Options

The following test options are available when using Steady Rate Sweep:

- Delay Before Test
- Analog Data Input

# Strain-Controlled Transient Test Methods

## Step Shear Rate

### Functional Description

Step Shear Rate is a steady transient test that applies a constant commanded shear rate for a selected time period. Up to four separate zones can be programmed, each with its own set of parameters (Figure 3-14). A maximum of 350 data points can be sampled in each zone. The interval between data points can be incremented either logarithmically or linearly. Within each zone the following are then set:

#### Shear Rate

Commanded shear rate in reciprocal seconds. Entering a Shear Rate of zero prevents actuator movement during data collection, allowing study of relaxation after steady shear.

#### Zone Time

Total time during which Shear Rate is commanded.

#### Direction

Direction specifies the rotational direction of the actuator for positive Shear Rate values.

Zone Number	1	2	3	4
Shear Rate [1/s]	-6.28	0.0	0.0	0.0
Zone Time [s or h:m:s]	100	0	0	0

Figure 3-14. Step Shear Rate Test Set-Up Form



## Suggested Uses

Step Shear Rate can be used to examine the following sample characteristics:

- Stress growth and relaxation at constant temperature
- Time required to reach steady-state flow behavior.
- Relaxation after steady shear (see Shear Rate in the description of Parameters)

## Test Options

The following test options are available when using Step Rate with the specified instrument types.

- Delay Before Test

# Stress Relaxation (Transient Step Strain)

## Functional Description

Stress Relaxation (transient step strain) monitors sample stress relaxation by taking measurements following a single upward or downward step of the motor to the selected strain. Four independent measurement "zones", with a maximum of 350 data points per zone, are available. The force response (relaxation profile or  $G(t)$ ) to the step strain is measured in either logarithmic or linear sampling mode.

### Log (Logarithmic)

Logarithmic sampling takes data at logarithmically incremented intervals. As an example, selecting 5 points per zone during a 100-second zone divides the difference of the endpoint times (logarithms) into five equally-spaced fractional exponents. Six data points are measured, in succession, at times determined by taking the antilogarithm of each exponent: 10, 15.9, 25.1, 39.8, 63.1, and 100 seconds.

### Linear

Linear sampling takes data at linearly incremented intervals. As an example, selecting 5 points per zone during a 100-second zone results in five data points measured at linear increments: 20, 40, 60, 80, and 100 seconds.

Select a strain, and direction, which is maintained throughout the entire test (Figure 3-15), and then a sample time (zone time) for each of the four zones. The times can be anywhere from 2.0 to  $1.6 \times 10^8$  seconds in length. Usually, the first zones are set at very short time periods (typically from 2.0 to 10 seconds), as most of the relaxation happens very quickly, with subsequent zones being set to longer times. Please note, that regardless of the field inputs, the fastest the instrument will take data is 1 data point every 3 msec.

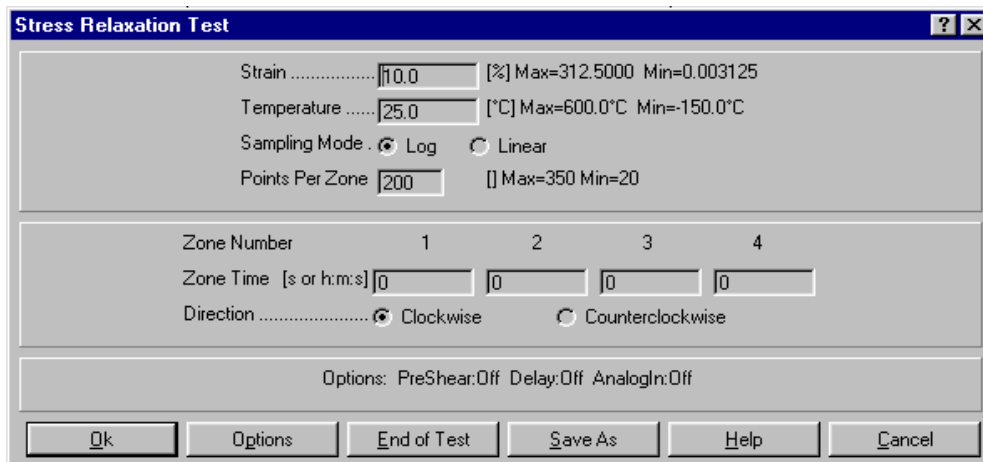


Figure 3-15. Stress Relaxation Test Set-up Screen

## Suggested Uses

Suggested uses of strain relaxation are as follows:

- Determination of time required for a sample material to relax after a deformation (as in sample material loading).
- Analysis of time dependent behavior of a sample.

## Test Options

- Delay Before Test
- Steady Preshear
- Analog Data Input

# Multiple Extension Mode

## Functional Description

Multiple Extension mode offers four zones in which a variety of separate extensional test types can be performed. The mode of extension can be based on linear rate, Hencky strain rate (related to the specimen geometry), force imposed to create the extension, or as a compressive Rim Shear mode (also known as squeeze flow).

The set-up screen for multiple extension mode (Figure 3-16) requires you to select a zone time for each zone. This is the time in seconds/minutes/hours over which the test type will occur in each zone. The extensional value units are dependent upon the test mode selected.

Several different test options are available in this mode. Any test type can be combined in any sequence during a multiple extension mode test. A fifth test type selection that is available is "End Test", which halts the series of extensional mode testing. Table 3-4 provides a summary of the different test types available and the uses of each of them.

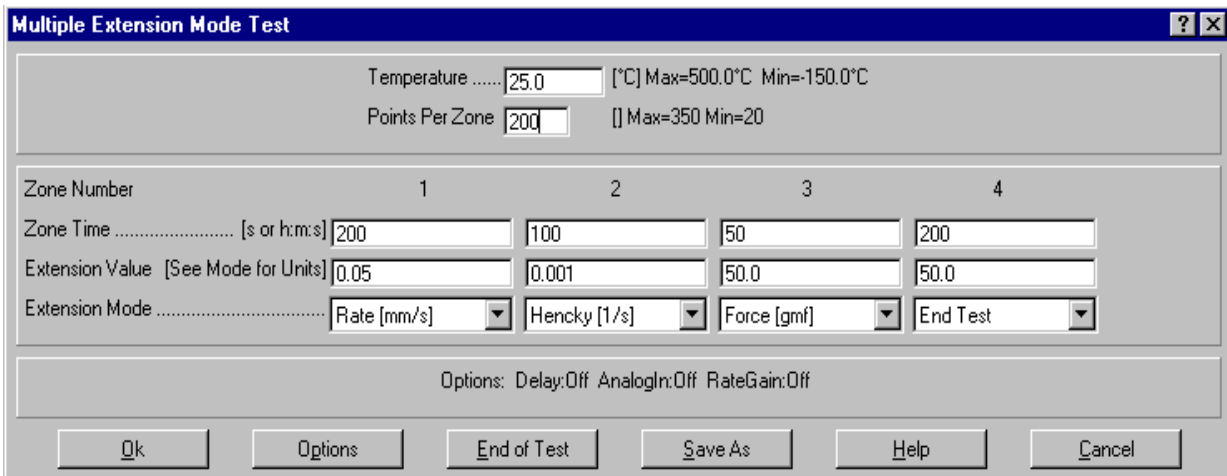


Figure 3-16. Multiple Extension Mode Test Set-up Screen

## Test Options

Multiple Extension Mode Test options are as follows:

- Delay Before Test
- Analog Data Input
- Motor Control Gain - Manual or Default (only for the Constant Force Mode)
  - Proportional Factor gain settings (manual or default of 1.0)
  - Integral Factor gain settings (manual or default of 0.1)

The motor control gain can be adjusted to change the response of the control loop controlling stress level in constant stress mode.

Table 3-4. Summary of Multiple Extension Mode Test Options.

<b>Extension Mode</b>	<b>Description</b>	<b>Usage</b>
Rate [mm/s]	Apply a constant rate of linear displacement. Extension rate in the sample is not constant due to change in sample length.	Can be used to determine the range of linear behavior in a material by plotting Force as a function of displacement.
Hencky [1/s]	Apply a constant rate of sample strain. Linear displacement rate is adjusted to maintain a constant sample strain rate.	Used to measure the extensional modulus and properties in samples.
Rim Shear [1/s]	Apply a constant shear rate to the sample in squeeze flow. Rate corresponds to the shear rate at the edge of the sample.	Measure the squeeze flow properties of a material.
Force [gmf]	Apply and maintain a constant force on the sample.	This can be used to provide information on the creep behavior of a material.
End Test	Used to indicate that a zone is not used and that the test should end.	---

# Arbitrary Waveshape Test

## Functional Description

The Arbitrary Waveshape test allows you to define the strain history used to deform the sample by supplying one or more equations for strain as a function of time. Up to four different equations, each with a specified time period can be used. There are also 4 "Zones" used for data collection, with each Zone capable of sampling over a time interval that is independent of the time specified for the waveform.

You should first define the time intervals for each of the data collection Zones (Figure 3-17), and the number of points (samples) to collect in each Zone. The second part of the form is used to enter equations for the strain as a function of time, as well as a "playback" time (Wave Time), which determines the period of time over which the equation is to be played back. Note that the strain is always given in strain units (not percent strain), and the limits (based on the current geometry) are given below the equation fields. The waveform cannot be built if the strain value exceeded the limit at any point.

If the sum of the wave times is less than the sum of the data collection times, then the motor will remain stationary once the entire wave has been played.

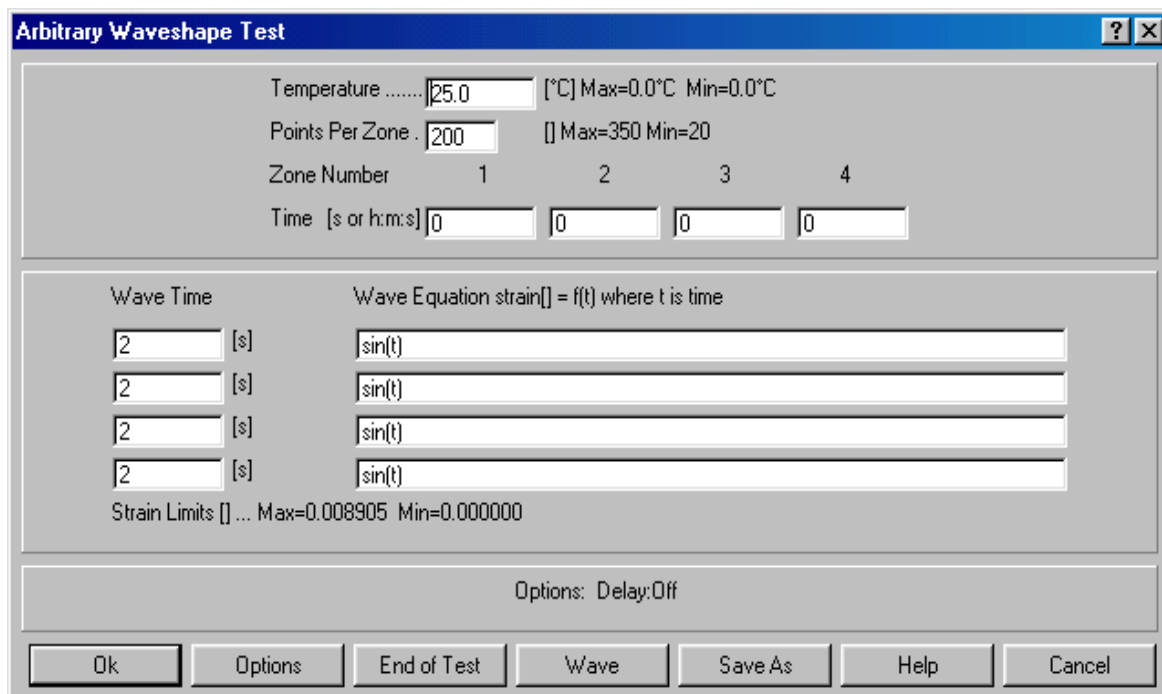


Figure 3-17. Arbitrary Waveshape Test Set-up Screen

In evaluating each equation, the time for each equation is based on the running time since the start of the first equation. For example, if two equations are used, each 2 seconds in length, the time variable is evaluated from 0-2 seconds for the first equation, and 2-4 seconds in the second. If there are discontinuities in the strain value between the end of one equation and the start of another, a jump in position will occur during measurement. If this is not desired, then the strain value should be contiguous from equation to equation in order to avoid such transients.

Once the strain equations and sampling times have been specified, the waveform must be sent to the instrument. The "Wave" button is used to compute the resulting waveform, display the results graphically, and provide the option to download directly into the instruments waveform memory. Any subsequent change to the equations or Wave Time fields requires recalculating and re-sending the waveform. The software keeps track of any new changes, and when the test is started, reminds you to resend the waveform if necessary.

The Zone 1 through Zone 4 times, as displayed on the host computer, refer to data collection time, and are independent of the wave playback times. That is, the waveforms will be played back, one immediately after the other, based on their entered Wave Times, and not on the data collection Zone times. It is possible to playback more than one waveform in a Zone, or play an equation across Zones. Once all the waveforms have been played, the motor will stop movement, but the software will continue to acquire data until the end of the last entered data collection Zone time.

Consider the following example:

Equation One Wave Time = 10 seconds; Equation Two Wave Time = 15 seconds  
Zone 1 acquisition time = 20 seconds; Zone 2 acquisition time = 10 seconds

Data will be collected in Zone 1 for the entire Equation One playback duration (Wave Time) of 10 seconds. As soon as Equation One finishes, Equation Two will start playing, with data still being collected in Zone 1. Ten seconds later (Equation Two still playing) data collection will end in Zone 1 and will data collection will start in Zone 2. After five more seconds, Equation Two will finish playing, and the motor will stop. Data will be collected for an additional 5 seconds in Zone 2, after which the test will end.

## Equation Syntax

Equations for Waveshape are entered as a function of time using the variable "t". Standard arithmetic operators can be used (+, -, \*, /, ^), as well as a variety of mathematical functions such as sin(), cos(), exp(), etc. Standard rules for operator precedence are used, and parenthesis may be used to change the order of evaluation. Table 3-5 contains a list of available functions.

## Test Options

- Delay Before Test
- Analog Data input

Table 3-5. Available functions for the Arbitrary Waveshape Test. Note: The arguments to trigonometric functions are in radians.

Operator	Mathematical Operation	Precedence
^	Exponentiation	Highest
*	Multiplication	
/	Division	
+	Addition	
-	Subtraction	Lowest

Functions	
sin( )	Sine
cos( )	Cosine
tan( )	Tangent
log( )	Common logarithm
ln( )	Natural logarithm
exp( )	base e raised to the x power
sinh( )	Hyperbolic sine
cosh( )	Hyperbolic cosine
asin( )	Arcsine
acos( )	Arccosine
atan( )	Arctangent
sqrt( )	Square root
abs( )	Absolute value



## Thixotropic Loop (Rate Ramp)

### Description

In each of up to four zones, Thixotropic Loop commands a steady shear rate that linearly accelerates or decelerates to a final shear rate. The time allotted to achieve the final shear rate is selectable in each zone, offering control of actuator acceleration (Figure 3-18).

In each zone the following parameters are set:

#### Final Shear Rate

Shear rate to which the initial rate is accelerated or decelerated. In the first zone, the initial rate is zero. In subsequent zones, the initial rate is the Final Shear Rate from the previous zone.

#### Zone Time

Total time allotted to achieve the Final Shear Rate.

#### Direction

Direction specifies the rotational direction of the actuator for positive Final Shear Rate values.

Zone Number	1	2	3	4
Final Shear Rate . [1/s]	0.0	0.0	0.0	0.0
Zone Time [s or h:m:s]	0	0	0	0

Figure 3-18. Thixotropic Loop Test Set-Up Form

### Suggested Uses

- Thixotropic Loop is used to determine material thixotropy, or hysteresis. This refers to the dependence of the material's response (as measured by shear stress or viscosity) to the previous strain rate history. This in turn, can be related to the build-up or breakdown of structure in complex fluids.

## Options

The following test option is available for use with Thixotropic Loop.

- Delay Before Test

# Torque/Normal Relaxation

## Functional Description

Torque/Normal Relaxation applies and maintains a single transient deformation (step strain). Data is then collected during each of four zones, the duration of each can be selected (Figure 3-19). Torque and normal force are monitored and reported during the test. This is in contrast to the Stress Relaxation experiment, which uses the same type of deformation, but monitors torque and strain instead of torque and normal force. The direction selected is the rotational direction of the actuator for positive Strain values. Data can be taken in either of two modes:

### Log (Logarithmic)

Logarithmic sampling takes data at logarithmically incremented intervals, resulting in equally spaced data points when plotted as a function of logarithmically-scaled time (the number of points taken is inversely proportional to zone time). As an example, selecting 5 points per zone during a 100 second zone divides the difference of the endpoint times (logarithms) into five equally-spaced fractional exponents. Six data points are measured, in succession, at times determined by taking the antilogarithm of each exponent: 10, 15.9, 25.1, 39.8, 63.1, and 100 seconds.

### Linear

Linear sampling takes data at linearly incremented intervals, resulting in equally spaced data points when plotted as a function of linearly-scaled time. This technique is useful for relatively short zone times, where linear time scaling is practical. As an example, selecting 5 points per zone during a 100-second zone results in five data points measured at linear increments: 20, 40, 60, 80, and 100 seconds.

The screenshot shows a software dialog box titled "Torque/Normal Relaxation Test". It has a standard Windows-style title bar with a question mark and close button. The main area contains several rows of controls: "Strain" with a text box containing "10.0" and units "[%] Max=312.5000 Min=0.003125"; "Temperature" with a text box containing "25.0" and units "[°C] Max=600.0°C Min=-150.0°C"; "Sampling Mode" with two radio buttons, "Log" being selected and "Linear" unselected; "Points Per Zone" with a text box containing "200" and "[Max=350 Min=20]"; a section for "Zone" settings with four columns for "Zone Number" (1, 2, 3, 4) and "Zone Time [s or h:m:s]" (all containing "0"); "Direction" with two radio buttons, "Clockwise" selected and "Counterclockwise" unselected; and "Options: PreShear:Off Delay:Off AnalogIn:Off". At the bottom, there is a row of buttons: "Ok", "Options", "End of Test", "Save As", "Help", and "Cancel".

Figure 3-19. Torque/Normal Relaxation Test Set-Up Form

## Suggested Uses

- Determination of time required for a sample material to relax after a deformation (as in sample material loading).
- Analysis of time dependent behavior of a sample.

## Options

The following test options are available when using Stress Relaxation:

- Delay Before Test
- Steady PreShear

# Force Gap Test

## Description

Executing the Force Gap test adjusts the sample gap to a specified value over a specified time. Data can be collected during the time that the gap is changing. The following parameters are set through the Force Gap Test Set-Up Screen (Figure 3-20).

### Force Sample Gap to \_\_\_\_\_

In this field, enter the sample gap that is desired at the end of the Force Gap test. The test concludes when this gap is reached, regardless of the Gap Adjustment Time (next item).

### Gap Adjustment Time

This field determines the rate at which the gap command changes. Enter the time period during which the gap will change from initial position (at the start of the test) to the gap specified in the Force Sample Gap to field.

### Save Test Data

When checked, saves data that have been collected while the gap was changing. The data are reported as gap (Instrument Gap Measurement) versus time.

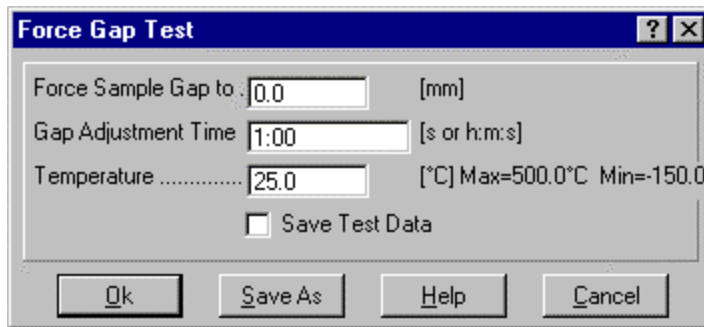


Figure 3-20. Force Gap Test Set-up Screen

## Suggested Uses

This test is useful during test sequences, as it can be inserted into the sequence to effect a controlled gap adjustment between tests.

This test is also good for sampling loading. It allows for documentable, consistent sample loading, and will minimize damage to, or axial force on a sample during loading.

## Test Options

There are no options available with this test.

# Steady Step Rate Temperature Ramp

## Description

Steady Rate Temperature Ramp monitors material stress and viscosity as a function of time at a series of specified shear rates within up to eight independently programmable zones. Within each zone, you can ramp temperature (at a selectable rate) upward or downward from the initial temperature (Figure 3-21). In each zone, specifying Log sampling takes the specified number of data points at logarithmically incremented intervals. Selecting Linear takes the specified number of data points at linearly incremented intervals. A maximum of 350 data points can be taken in each zone.

In Each Zone the Final Temp is the Temperature at which the instrument stops ramping temperature while in the respective zone. The Ramp Rate is the Rate of thermal change that the sample material undergoes during the test. Orchestrator reports the difference between the Initial Temperature and Final Temperature setpoints, divided by the Ramp Rate that you specified as the computed Ramp time.

Ramp direction is set using the Initial and Final Temperatures in a zone. The Ramp Rate is in units of °C per minute (°C/min.), and the entry field accepts any value within the range of the instrument, with a display resolution of 0.1°C. If temperature control is not needed, and the deformation is the only desired change, the Ramp Rate should be set to 0°C/min. A Ramp Rate of 0°C/min. makes the test a time sweep; hence, this test is sometimes referred to as a time/cure sweep.

Following the Ramp, the Soak Time After Ramp is the time period during which temperature is held at the Final Temperature. Orchestrator reports the total time required to run the test, including all zones and soak times as the Computed Test Duration.

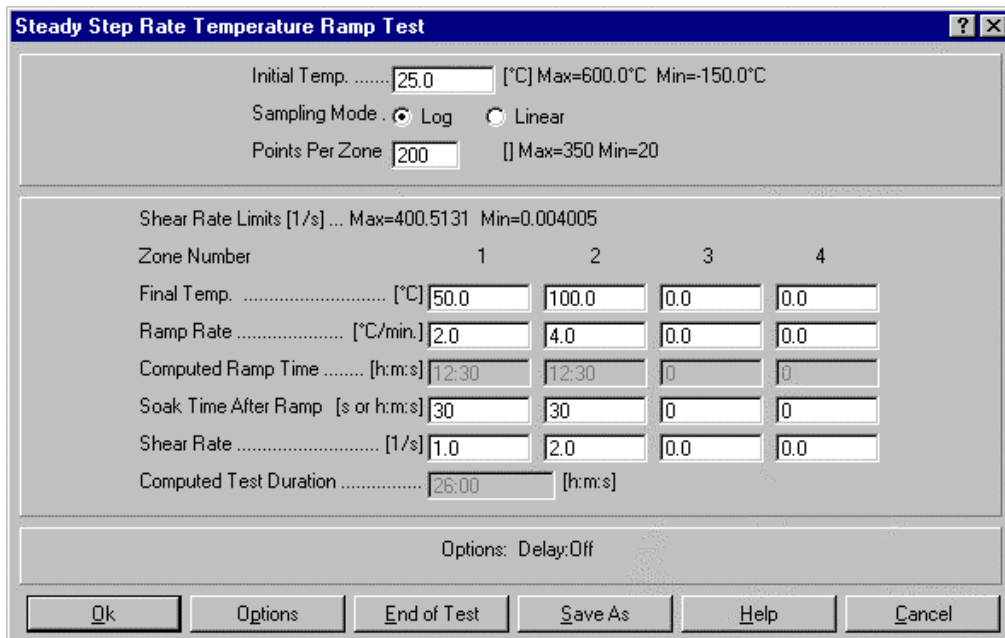


Figure 3-21. Steady Step Rate Temperature Ramp Set-Up Screen

## Suggested Uses

Steady Step rate Temperature Ramp can be used to examine the following:

- Process simulation
- Transient material response to changing shear rates and temperatures.

## Options

The following test option is available when using Steady Step Rate Temperature Ramp:

- Delay Before Test

# Stress-Controlled Transient Test Methods

## Constant Stress Test

### Functional Description

Constant Stress is a transient test that applies a stress, in a selected direction for a specified time period, in up to two zones. In each zone, rate is varied to maintain stress until either a strain or time limit is reached, at which time zero stress is commanded.

When setting up the Constant Stress test, the desired temperature to conduct the test and the sample estimated viscosity (calculated by dividing the shear stress by the shear rate) are first entered (Figure 3-22). Then the Maximum Allowed Strain, which is the maximum displacement of the actuator throughout the test, is entered. When either the maximum allowed strain or time (see next item) is reached, zero rate (and zero stress) is commanded. The estimated viscosity value is used to modify the gain term in the closed-loop control algorithm, which adjusts the motor's rotational rate to generate the desired stress level.

For each zone the following are entered:

#### Stress

Commanded stress, which is maintained by varying shear rate. Positive Stress results in clockwise rotation. Negative Stress results in counterclockwise rotation.

#### Time

Duration that shear stress is commanded, unless Max Allowed Strain (see previous item) is reached first. When either Time or Max Allowed Strain is reached, zero rate (and zero stress) is commanded.

Temperature	25.0	[°C]	Max=600.0°C	Min=-150.0°C	
Estimated Viscosity	3.00e+05	[Pa·s]			
Max Allowed Strain	1.00e+06	[%]			
Points Per Zone	200		Max=350	Min=20	
Stress Limits [Pa]	Max=±63929.42 Min=±6.392942				
Zone Number	1	2			
Stress [Pa]	300.0	0.0			
Zone Time [s or h:m:s]	30	0			
Options:	Delay:Off MotorPID:On				
Ok	Options	End of Test	Save As	Help	Cancel

Figure 3-22. Constant Stress Test Set-Up Screen



## Suggested Uses

This test is used to measure the creep response of materials, which provides transient information of the measured strain as a function of the commanded stress.

## Options

The following test options can be selected for use with the Constant Stress test:

- Delay Before Test
- Steady PreShear
- Motor PID

# Stress Ramp Test

## Description

Stress Ramp commands a steady stress level from an initial to a final stress at a selectable, linear rate that is based upon zone time. Positive Final Stress values result in clockwise rotation of the actuator (stress head); negative values result in counterclockwise rotation. In each zone the data can be sampled at a logarithmic or linear interval (Figure 3-23).

Logarithmic sampling takes data at logarithmically incremented intervals, resulting in equally spaced data points when plotted as a function of logarithmically-scaled time (the number of points taken is inversely proportional to zone time). Linear sampling takes data at linearly incremented intervals, resulting in equally spaced data points when plotted as a function of linearly-scaled time. This technique is useful for relatively short zone times, where linear time scaling is practical. The estimated viscosity value is used to modify the gain term in the closed-loop control algorithm, which adjusts the motor's rotational rate to generate the desired stress level.

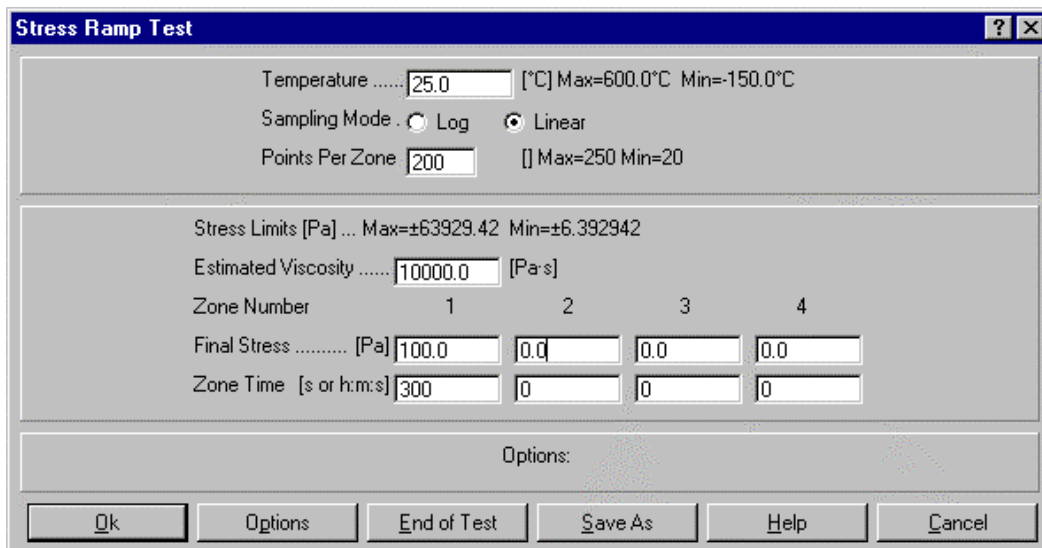


Figure 3-23. Stress Ramp Test Set-Up Screen

## Suggested Use

Stress Ramp is used to determine material hysteresis by deforming the material by linearly accelerating or decelerating the shear stress command. The test is similar to the thixotropic loop test available on Shear Strain-Controlled instruments.

## Options

The following test options are available for use with Stress Ramp:

- Delay Before Test

# Test Options

This section describes the following test options that you can select while programming selected dynamic tests:

- Steady PreShear
- Delay Before Test
- Auto Tension Adjustment
- Analog Data Input
- Auto Strain Adjustment
- Measurement Options

## Steady PreShear

Steady PreShear allows you to subject the sample to a steady shear deformation prior to the start of a dynamic test. If the Delay Before Test option is selected, Steady PreShear occurs prior to the delay. No data are taken while the pre-shear is being applied.

The following parameters are set through the Steady PreShear Set-Up Screen (Figure 3-24):

### PreShear Rate

Shear rate of the PreShear stress. Positive PreShear Rates result in clockwise actuator rotation. Negative PreShear Rates result in counterclockwise actuator rotation.

### PreShear Time

The length of time the PreShear is applied.

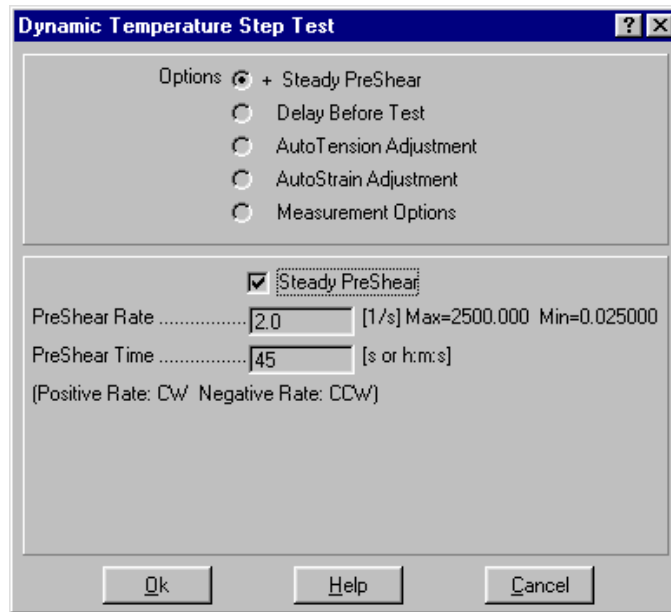


Figure 3-24. Steady PreShear Set-Up Screen

# Delay Before Test

## Manual Delay

A manually entered "Delay Before Test" is the time period between the start of the test and the first measurement (1 through 65,000 seconds). The time period selected in "Delay Before Test" (Figure 3-25) allows the instrument to equilibrate, or the sample to relax, prior to imposing the deformation.

## Apply AutoTension

The "Apply AutoTension at the End of Delay" check box determines the point at which AutoTension is applied. If left unchecked, AutoTension is applied at the start of the Delay period. Checking the box causes it to not be applied until the delay period has ended.

## Automatically Start Test When On Temperature

When checked, the start of the test is delayed until the Environmental Control System has stabilized at the Commanded Temperature.

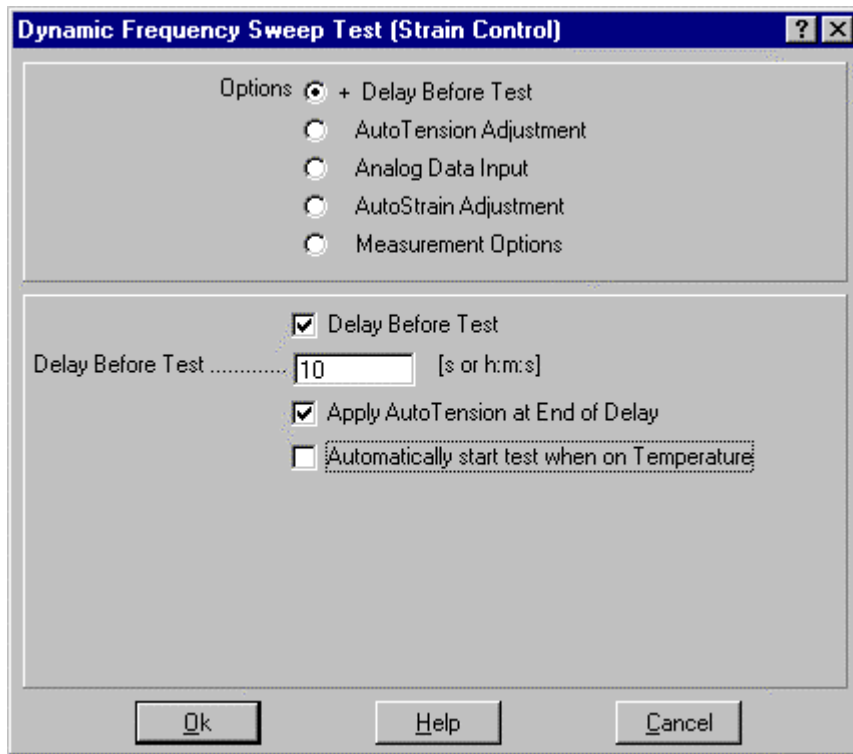


Figure 3-25. Delay Before Test Options Set-Up Screen

## AutoTension Adjustment

AutoTension maintains a specified axial static force on the sample. AutoTension can apply tensile force to keep the sample taut (prevent sample buckling), or compressive force to maintain a compressive load and prevent loss of test tool contact. During dynamic testing using the Torsion Rectangular test tool, AutoTension can be used to compensate for the change in sample length that occurs as a result of thermal expansion. Using AutoTension with tools such as Parallel Plate can prevent loss of contact between sample and tool.

Following is a description of the AutoTension parameters that must be set in the AutoTension Adjustment Set-Up Screen (Fig 3-26).

### AutoTension Direction

When Tension is selected, tensile static force is applied. When Compression is selected, compressive static force is applied.

### Initial Static Force

This is defined as the static force that is maintained throughout the test.

### AutoTension Sensitivity

Minimum change in normal force that results in an adjustment to maintain the Initial Static Force.

### Switch AutoTension to Programmed Extension When Sample Modulus....

When sample modulus decreases below the value entered in this field, the last valid coefficient of thermal expansion ( $\alpha$ ) of the sample is used to determine an AutoTension static force that best maintains the sample length. The coefficient of thermal expansion ( $\alpha$ ) is given by:

$$\alpha = \frac{\Delta L / L}{\Delta T}$$

where:  $\Delta L$  = change in sample length  
 $L$  = sample length  
 $\Delta T$  = change in sample temperature

This feature is useful when running temperature "sweeps", to prevent excess sample stretching with increasing temperature. It is important to apply this adjustment when sampling through the glass transition point, where there will be a significant, rapid drop in  $G'$ . Without this adjustment, the now "softer" sample (beyond the glass transition point) would rapidly stretch and ultimately be pulled part. A test run may be necessary to determine where the glass transition point occurs before setting this value.

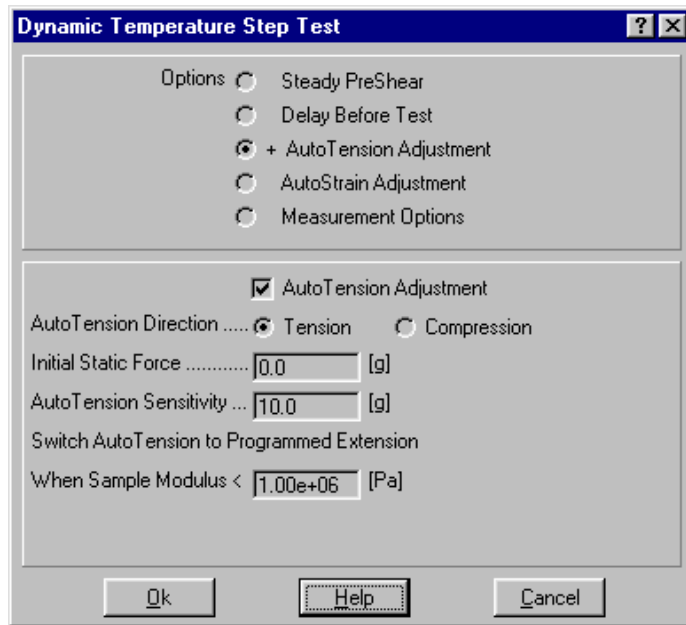


Figure 3-26. AutoTension Adjustment Set-Up Screen

## Analog Data Input

Analog Data Input allows you to apply an external  $\pm 10$  VDC analog input signal to the Analog Input BNC (ANALOG 1 IN) receptacle on the Test Station Rear Panel, and measure, and record with the rest of the data, the input signal during any test. Data are sampled at 1 Hz, and only one measurement is recorded for each data point.

Apply an external  $\pm 10$  VDC input signal, then check the Analog Data Collection checkbox (Figure 3-27). During the test, Orchestrator reports the analog input as Analog Input Data, which can be displayed in the spreadsheet, plot, or as an on-line parameter. The reported values are normalized by dividing input voltage by 10. Thus, an input voltage of +10 volts is recorded in the data as +1.00.

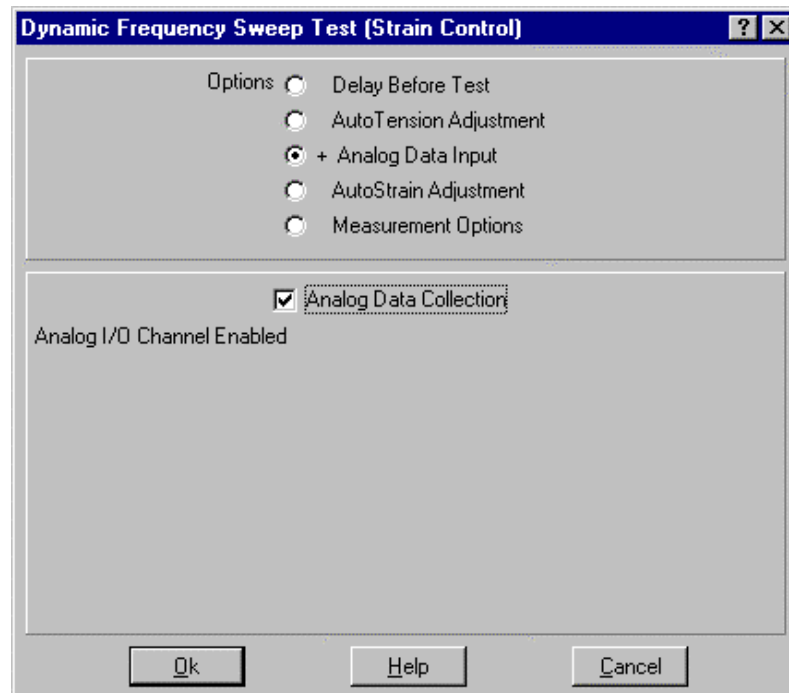


Figure 3-27. Analog Data Input Set-Up Screen

## AutoStrain

AutoStrain is used to prevent the dynamic force signal from going above or below the specified force limits of the transducer. When using AutoStrain control, the actual sample strain may differ from that commanded.

Following is a description of AutoStrain parameters that must be set (Figure 3-28):

### Maximum Applied Strain \_\_\_\_\_

This value represents the maximum allowed strain to be used in the AutoStrain adjustment. The strain amplitude will never exceed this value irrespective of the measured force.

### Maximum Allowed Force \_\_\_\_\_

When the dynamic force rises above this value, strain is decreased. This should be set this to the maximum dynamic force that you wish to maintain during a measurement.

### Minimum Allowed Force \_\_\_\_\_

When the dynamic force drops below this value, strain is increased. This should be set to the minimum dynamic force that you wish to maintain during a measurement.

### Strain Adjustment \_\_\_\_\_

This is the percentile by which strain is increased or decreased when the measured dynamic force is below the entered Minimum Allowed Force or above the entered Maximum Allowed Force. The percentage of commanded strain entered depends upon how fast the dynamic force is decreasing between measurements.

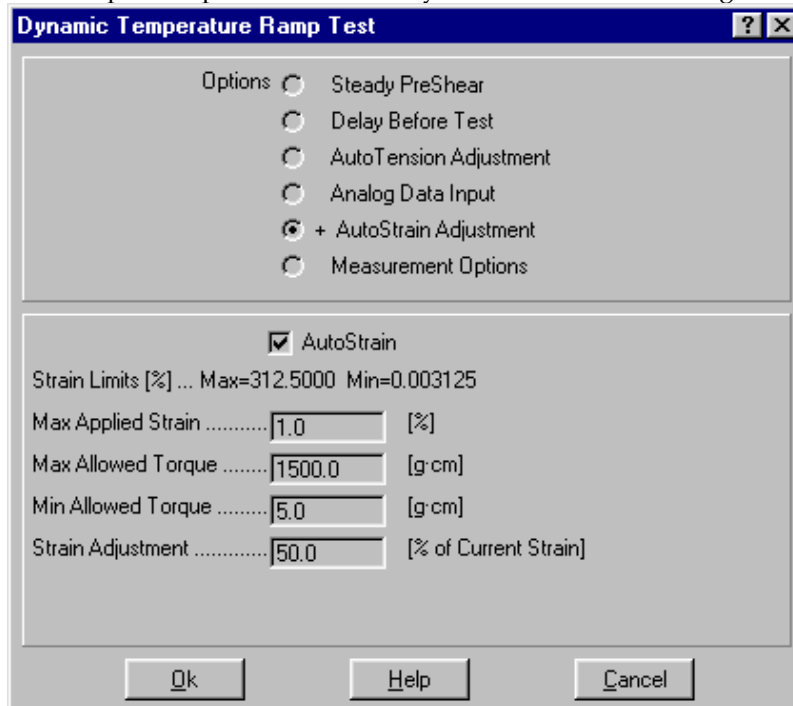


Figure 3-28. AutoStrain Set-Up Screen



## Measurement Options

The Measurement Options in a test can control the delay settings. The Default Delay setting gives 0.5 cycles and 3 seconds (whichever is longer) delay before data collection. For some samples this may need to be increased to allow the material to reach a steady-state response. Manual settings can be adjusted as shown in Figure 3-29.

### Correlation Delay

Correlation delay is the time period between the start of sample deformation and the first measurement in a dynamic test. During this period the instrument does not collect data. Note that data collection is delayed, not motor movement. Correlation delay allows the sample to achieve equilibrium with the test conditions before the measurement is taken, and allows development of the phase angle ( $\delta$ ) from the start of stress and strain sine waves. Internally, the instrument computes the correlation delay in seconds, with a minimum increment of 0.1 second and a minimum time period of 0.2 second. The number of cycles to which this corresponds varies depending upon the frequency. The maximum allowable time period is 65,000 seconds.

### One Cycle Correlation

The One Cycle Correlation option speeds the test by commanding the control computer to use data measured over one cycle, regardless of test frequency. Normally, at frequencies above 2 rad/sec data are collected over multiple cycles and averaged. One Cycle Correlation forces measurements to be made with only one cycle worth of data, saving the time that would be required for the subsequent cycles. One Cycle Correlation is useful for applications requiring fast measurements such as sample curing at high frequencies. The disadvantage is that, due to the absence of data averaging, resulting data points may contain more noise than data gathered over many cycles.

### Strain Amplitude Control

Measurement does not begin until the strain that you command during a test is within a certain percentage of the strain actually applied to the sample. This feature allows you to specify this percentage:

#### Default

Does not use strain amplitude control. The actual sample strain is simply recorded.

#### Adjustable

Measurement begins when sample strain is within the entered percentage of initial commanded strain. This will increase the test time as the motor movement increases to reach the desired sample strain.

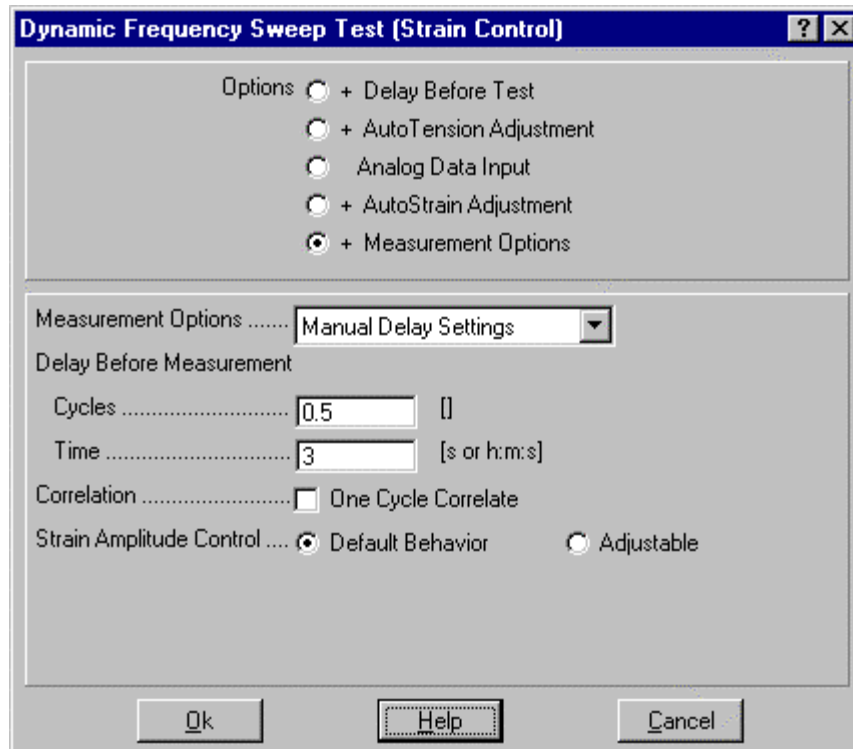


Figure 3-29. Measurement Options Set-up Screen.

---

---

# Chapter 4

## Test Geometries and Formulas

### Introduction

This chapter provides information on the various test geometries and formulas that are used with the ARES instrument. The following material is covered:

- General Test Tool Information
- General Test Tool Installation
- Zeroing and Setting the Gap
- Specific Test Tool Geometries
- Test Formulas

### General Test Tool Information

#### *General Recommendations for Geometry Selection*

Although the physical properties of the sample generally dictate appropriate sample geometry, it is sometimes possible to test a given sample using more than one geometry. Ideally, the test results should be identical in the different geometries. However, there exist experimental limitations that may make testing in one geometry preferable to testing in another. Additionally, factors such as anisotropy and differences in strain dependence may yield inconsistent results for different geometries.

Recommendations for selection of a geometry based upon sample type are as follows:

#### Thin Films or Fibers

Test thin films such as magnetic recording tape, or fibers, using the fiber/film tool. Enter the test geometry dimensions into the appropriate Tension geometry screen.

#### Fluids, Suspensions and Emulsions

Low viscosity fluids, or suspensions of limited stability can be tested using either the Couette or Double Wall Couette geometry. Higher viscosity fluids, and thicker suspensions and emulsions can be tested using parallel plates, or cone and plate geometries.

#### Solid Samples, Including Thermosets, Thermoplastics, and Elastomers

These materials can be tested using the Torsion Rectangular tool. Several inserts are available to accommodate a variety of sample thicknesses.

## Polymer Melts and Soft Solids

Melts can be tested using the parallel plate or cone and plate geometries.

## Thermosetting Resins and other Curing Studies

These materials are best tested in a parallel plate tool. Disposable plates are available for curing studies, or other tool destructive materials.

## *Testing Limits and Compliance*

### Definition of Compliance and Stiffness

For this discussion, compliance is defined as displacement, in radians, per gram-centimeter of applied torque. Stiffness is the reciprocal of compliance. Transducer compliance is the transducer shaft displacement resulting from torque applied to the transducer. Shaft displacement is measured by a position sensor on the transducer shaft. Sample compliance is sample displacement resulting from force applied to the sample.

Because the transducer is not an infinitely stiff device, both the transducer and sample exhibit compliance. Since the transducer is being deformed along with the sample, some of the strain that is commanded deforms the sample, and some of the strain deforms the transducer. This leads to errors in sample moduli, which becomes larger as sample stiffness increases.

ARES utilizes an on-line hardware correction scheme to adjust for transducer compliance. The system determines sample deformation (strain) by taking the difference between the (measured) motor and transducer displacement signals. The measurement is sensitive to limits in strain resolution, as well as variations in motor, and transducer, calibration and linearity. Under "ideal" conditions, the sample deformation is relatively large, and as such, the transducer displacement is much smaller than the motor displacement applying the torque. The difference between the two deformations (which is used to obtain sample displacement) is therefore a large number, and the relative error associated with the measurement is small. However, this error becomes significant when very stiff samples are tested, and the transducer displacement becomes close to the motor displacement. In this case, because the difference between the two displacements is small, the resulting relative error is large and of similar magnitude as the measurement.

If the measured strain value is significantly smaller than the commanded strain value, the data are likely affected by transducer compliance. **As a practical guideline, measured sample strain should be at least 30% the commanded strain.** Although measurements can be taken below these limits, you are cautioned that accuracy may be affected. Measurements that are affected by transducer compliance typically report modulus values that are lower than the true modulus. One method of determining if transducer compliance is affecting the data is to switch to a different geometry and compare the results to the first tests. If the data are unaffected by compliance, the results from the two geometries should be nearly identical.

Sample compliance, or stiffness, is related to both the modulus and geometry of the sample. Since the modulus is fixed, the sample dimensions are normally adjusted (to make the sample less stiff), or the geometry is changed altogether, to obtain the desired sample compliance. It is critical that the sample compliance is within the operational range of the instrument otherwise inconsistent or incorrect results will be obtained.

## Determination of Operational Range

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using a specific geometry. For each geometry there are specific factors that will affect the operating range for that geometry. Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor

To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where:  $K_\tau$  = Stress Constant for the specific geometry

$K_\gamma$  = Strain Constant for the specific geometry and C is computed from the following:

Transducer	C for $G^*$ MAXIMUM		C for $G^*$ MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06$ (rad/g•cm)	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 4.9 e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
2K STD 10K STD	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
100 FRT 200 FRT 100 FRTN1 200 FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	for $\omega=100$ ; $J = 2.60e-05$ for $\omega=10$ ; $J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
	$C = \frac{M_{\max}}{\theta_{\min}}$	for $\omega < 10$ ; (see note below)		

NOTE: The values for  $M$  (g•cm) and  $\theta$  (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.

To determine the maximum or minimum complex viscosity  $\eta^*$  that can be measured at a given frequency, use the following formula:

$$\eta^* = \frac{G^*}{\omega} \quad (4-2)$$

where:  $\eta^*$  = Complex viscosity (Poise)  
 $G^*$  = Complex Modulus (dynes/cm<sup>2</sup>)  
 $\omega$  = Frequency (rad/sec)

Using a spreadsheet application such as Microsoft® Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate  $G^*$  MAXIMUM and  $G^*$  MINIMUM using equation 4-1.
2. Substitute the  $G^*$  MAXIMUM value into equation 4-2, then determine the upper limit of operation by calculating  $\eta^*$  at each  $\omega$  ( $\omega$  values chosen to be from the lowest to highest frequencies within the transducer operating range).
3. Substitute the  $G^*$  MINIMUM value into equation 4-2, then determine the lower limit of operation by calculating  $\eta^*$  at each  $\omega$ .
4. Generate an X-Y scatter plot of complex viscosity  $\eta^*$  (Y axis) versus frequency  $\omega$  (X axis).

The region between the upper and lower limits of operation is the range of complex viscosity that can be tested. Please note that the values for  $J$  above are nominal values, and will vary slightly between transducers. Accordingly (and per good standard practice) care should be taken (to ensure the data are valid) when testing near the upper or lower limits for a given system.

## *Possible Sources and Causes for Error*

This section contains information describing errors that can occur as a result of tool and sample limitations.

### Discrepancies in Sample Geometry

The force generated by a sample for a given amount of strain is a function of the material's modulus and the size of the sample. Therefore, discrepancies in sample dimensions will obviously affect the accuracy of test results. Referring to the equations for each geometries strain and stress constants, note the dependence of compliance on each of the dimensions. Due to this dependence, a small error in sample dimension may propagate into a large error in compliance, and therefore modulus.

### Testing outside the Linear Region

Most of the tests available are designed to be conducted within the samples linear region. Conducting tests outside the linear region may result in erratic or incorrect data. It is critical that for a given material the linear region is determined first, and then subsequent test performed based on the findings. To determine a materials linear region typically a Dynamic Strain Sweep is run as a preliminary test.

## Temperature Variations

Changes in sample temperature during measurement can have a significant effect on the measured dynamic data, particularly in the loss component of stress, and in  $\tan \delta$ . As a result, temperature steps (where temperature is held constant during a measurement) may yield better  $\tan \delta$  resolution when studying the temperature dependence of a material, as opposed to a temperature ramp, where temperature is changing during a measurement. Also, make sure that sufficient equilibration time is provided for the entire sample to reach the test temperature.

Some samples may undergo relatively large dimensional fluctuations as a result of temperature changes. AutoTension or AutoStrain may be necessary to adjust for temperature related changes.

## Other Factors

Other factors that will cause errors include sample edge failure, sample buckling, and sample slippage or clamping problems. Care should be taken to load the sample properly, determine its proper testing range, and, when needed, apply AutoTension correctly.

# General Test Tool Installation

---



**WARNING:** If this instrument is used in a manner not intended or specified in this manual, the protection provided by the instrument may be impaired.

---

## *Upper Tool Installation*

To install an upper tool, loosen the knob on the anvil (Figure 4-1) and insert the tool into the anvil, pulling apart the retainers if necessary. Tighten the knob. **Hand tighten the knobs - do not over-torque them.**

## *Lower Tool Installation: Motor Mount (Oven or Ambient)*

To install a lower tool onto the motor, refer to Figure 4-2 while performing the following steps:

1. If the tool accepts a PRT, install the tool PRT as follows:
  - a. Place (do not push) the plug onto the electrical jack mounted in the motor anvil.
  - b. While applying light downward force, rotate the tool PRT until it slips into place, indicating that the PRT has aligned with the keyway in the electrical jack. When properly installed, Orchestrator will indicate ambient temperature.
2. Loosen the knob on the anvil and insert the tool into the anvil, pulling apart the retainers if necessary. Hand-tighten the knob (do not over-torque).

## *Lower Tool Installation: Fluid Bath Mount*

To install a lower tool into either of the Fluids Baths, please refer to Chapter 2 for specific installation instructions. For either bath, the correct lower PRT should be inserted first, followed by the tool.



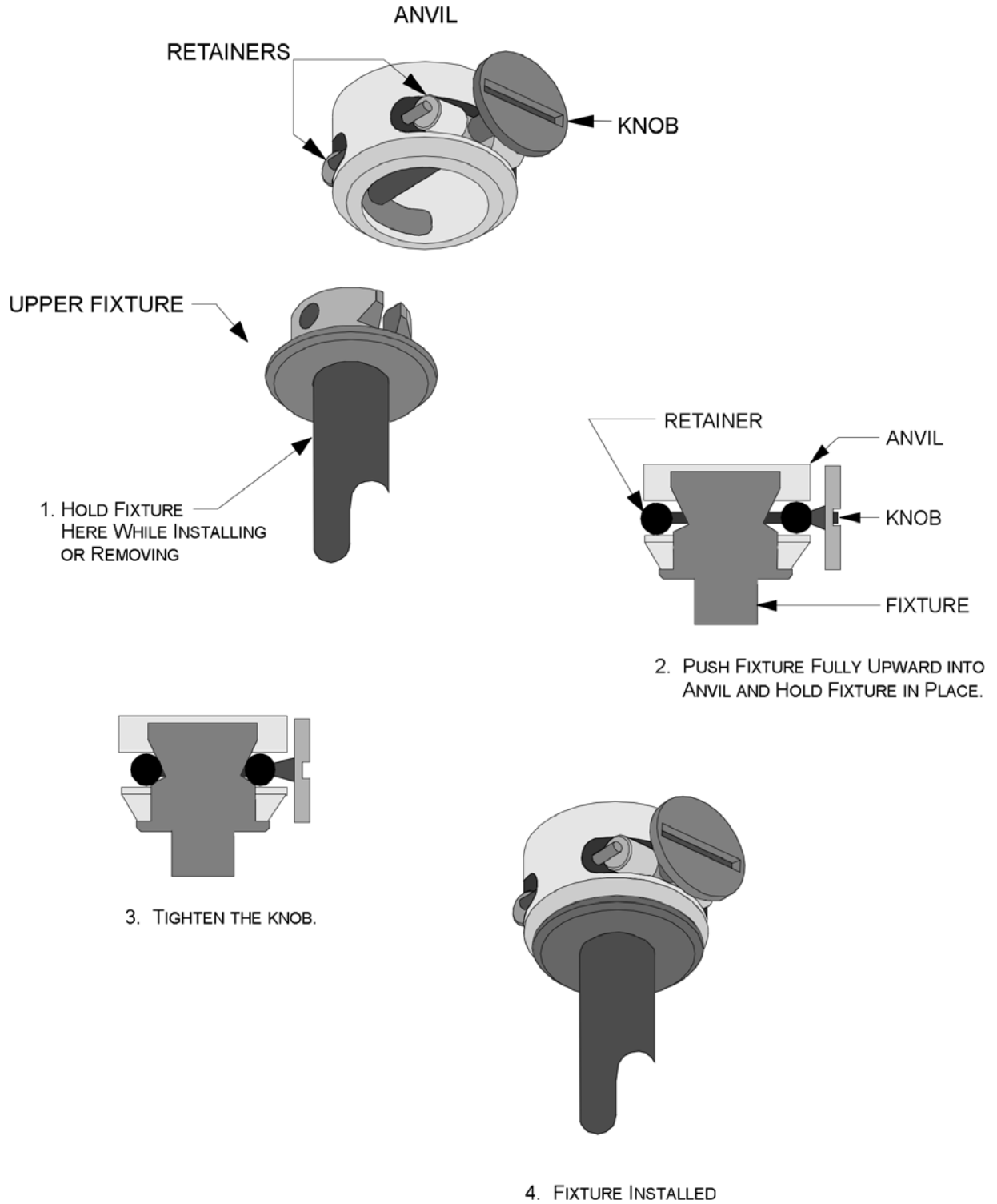


Figure 4-1. Upper Tool Installation

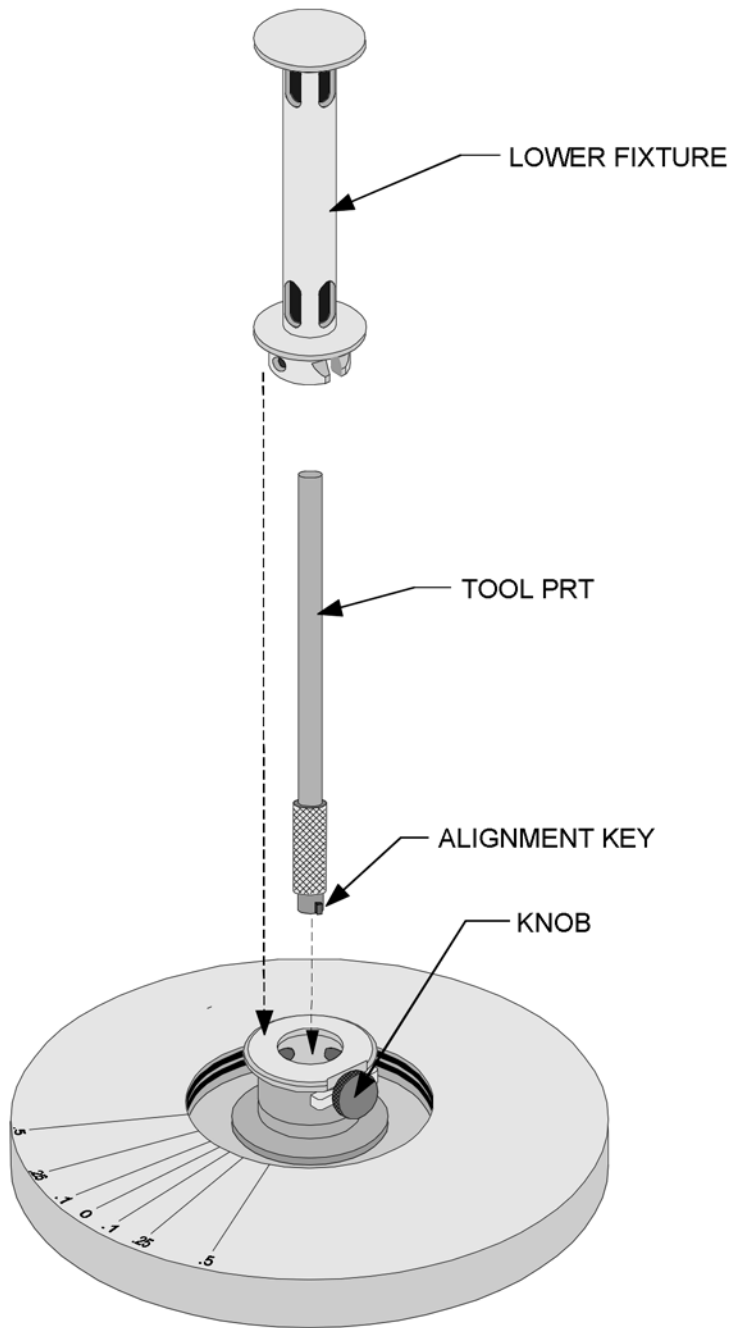


Figure 4-2. Lower Tool Installation, Motor Mount

# Setting the Gap

Zeroing the gap between upper and lower test tools is a prerequisite to setting the gap during sample loading. Zeroing the gap requires lowering the stage until the upper and lower tools touch, then setting the GAP value to read zero millimeters. After that, moving the stage results in an accurate gap indication as displayed by the GAP value.

The following procedures are general for all test tools. Specific details regarding each tool are described under that tool's individual section. Two methods are available for zeroing and setting the gap: Manual Zero and Auto Zero. For most applications, the Auto Zero method is recommended. The *Set Gap/Instrument Control* function in Orchestrator is used to control the stage movement when zeroing and setting the gap.

If testing is to be done at temperatures other than ambient, install the tool and then adjust the temperature to the desired initial value. Allow the tools to reach thermal equilibrium before zeroing and setting the gap.

## Manual Zero

To manually zero and set the gap between upper and lower tools, perform the following steps:

1. Ensure that the upper and lower tools are clean and install them.
2. Select the Set Gap/Instrument Control function. The Gap/Instrument Control Panel is displayed.
3. Zero the Torque and Normal Force by pressing the "Offset Torque To Zero" and "Offset Force To Zero" buttons.
4. Using the slew (rapid) mode of the Manual Stage Control, lower the stage until the distance between upper and lower tools is about 0.5 mm.
5. Using the step (slow) mode of the Stage Control, lower the stage until the Normal Force (indicated on the *Set Gap/Instrument Control Panel*) **just begins** to deflect from zero, indicating that the tools are in contact.
6. Zero the Gap value by pressing the "Zero Indicator" button under the displayed Gap.
7. Raise the stage to a height that allows the sample to be loaded.

The sample can now be loaded. When the stage is lowered, the *Set Gap/Instrument Control Panel* displays the correct distance between tools.

## Automatic Zeroing and Gap Setting

The *Set Gap/Instrument Control Panel* function allows you to automatically zero and set the gap between upper and lower tools.

### Zeroing the Gap

Establish a zero reference gap between Upper and Lower tools as follows:

1. Ensure that a sample is not loaded and the upper and lower tools are clean.
2. Using the Manual Stage Control, lower the stage to achieve a Gap of about 1 mm (as judged visually).
3. Select the *Set Gap/Instrument Control Panel* function. The *Set Gap/Instrument Control Panel* dialog box is displayed.
4. Zero the Torque and Normal Force by pressing the "Offset Torque To Zero" and "Offset Force To Zero" buttons.
5. Click "Zero Tool". An information form is displayed showing the duration (Elapsed Time) of the zeroing operation. The present gap is displayed in the Current Gap field, which updates at one-second intervals. The following events occur during the zeroing operation:
  - a. The stage lowers to achieve contact between tools.
  - b. Following contact, the Gap display is zeroed.
6. In preparation for loading the sample, raise the stage to maximum height by clicking Send to Top.

Remain in the Gap Control Panel and proceed to the next topic, "Setting the Gap."

### Setting the Gap

After zeroing the Gap, set it as follows:

1. Place the sample onto the lower tool.
2. Enter the following information:
  - a. Commanded Gap Position - Enter the desired Gap in millimeters.
  - b. Max Allowed Force - Enter the maximum Normal Force (in grams-force) that will be generated while the sample is being compressed during the gap setting operation. When Normal Force exceeds this value, the stage stops descending until Normal Force drops below this value. Stage movement then resumes.
3. Click Set Gap. An information form appears, indicating the time elapsed since the button was clicked. The Stage should descend relatively quickly (typically 5 mm/sec) until the Upper Tool is 3 mm from the Commanded Gap Position, at which time it will slow its rate of descent until the Gap is achieved. The gap is set when the information form is no longer displayed.

## Comments Concerning the Gap Control Panel

### Enabling the Gap Control Panel

The *Gap Control Panel* function can only operate if the Stepper Motor and Remote Gap Monitoring options are enabled. These options have been enabled at our manufacturing facility, and should require no modification. However, for your reference, these options are set by using the *Instrument Configuration* function located under the *Service* function of the *Utilities* pull-down menu. Set the "Stepper or Linear Motor" and "Remote Gap Monitoring" options to yes, as shown in Figure 4-3.

## Read Test Tool Gap Checkbox

The "Read Test Fixture Gap" checkbox is located in the form used to edit a specific geometry (accessed by clicking the "Edit Geometry" button in the *Edit/Start Test* dialog box). When the checkbox is selected, the instrument reads (and stores) the actual gap immediately prior to the start of a test. It then rechecks strain error limits using the actual gap (overriding the gap entered in the specific geometry form). Out-of-range limits are then reported.

For Stored Geometries, the "Read Test Fixture Gap" checkbox is displayed only if you saved a Stored Geometry while the "Remote Gap Monitoring" option was enabled (using the *Instrument Configuration* function).

## Displaying the Instrument Gap

The gap read from the instrument can be displayed as the online parameter CurrGap.

## Max Allowed Force While Setting the Gap

The "Max Allowed Force" option, when setting the gap, provides two benefits. First, it allows an operator to set the gap in a repeatable, documentable way. Loading the sample the same way each time leads to more reproducible results between different operators, or with different geometries. Additionally, it helps avoid internal pre-stressing a sample during loading, which again, could lead to erratic or inconsistent results. This feature is also helpful when loading soft samples that may squeeze out from between the test tool if too much force is applied.

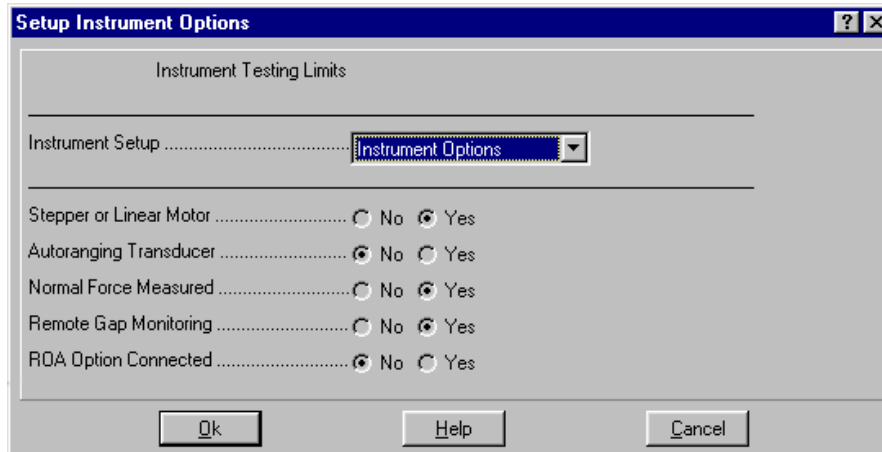


Figure 4-3. *Instrument Configuration Function* - "Instrument Options". Set the "Stepper or Linear Motor" and "Remote Gap Monitoring" options to yes to enable the *Gap Control Panel* functions.

# Test Tool Installation and Sample Loading

## *General Guidelines*

The next several sections contain specific procedures to install the test tools used in each geometry. Specific sample loading guidelines are given for each tool. However, the following general sample loading guidelines pertain to all tools:

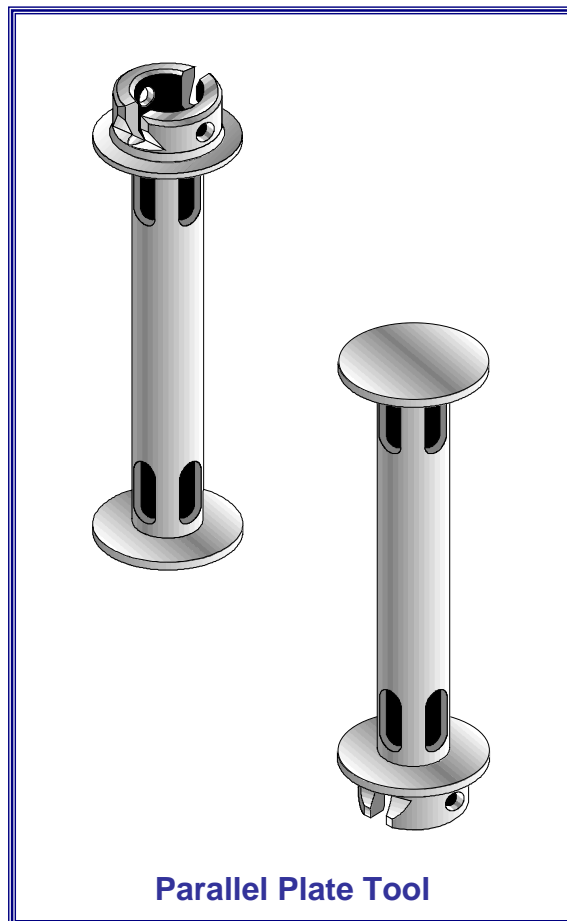
- Make sure the tools are clean and free from damage.
- Install the tools correctly as described. In general the tools should be easy to install. Having to use undue force is a sign that something is wrong. Stop and ascertain the problem before continuing.
- Make sure the tools align with each other properly and that the Gap is properly zeroed.
- When using the oven close the oven door carefully to ensure that the sample or tool does not interfere with the oven door.

While loading the sample onto the tool, ensure that the sample is centered, as well as is possible, between the tool mounting surfaces. Off-center loading may cause misalignment of the transducer/motor shafts. Additionally, misalignment may also affect the accuracy of the data. If misalignment does occur, the sample should be removed from the tool and carefully reloaded.

When testing at temperatures below ambient temperature (or the temperature at which the sample was loaded), the clamps (on some tools) may loosen as they cool. This is due to difference in the thermal expansion coefficients of the sample and that of the tools. Immediately prior to initiating testing at the lowest test temperature, you may wish to open the oven door and verify that the screws securing the clamps are tight, taking care not to touch any surface of the oven or tools which may be at dangerous cryogenic temperatures.

## Parallel Plates

<b>Strain Constant</b> $K_{\gamma} = \frac{R}{H}$	<b>Stress Constant</b> $K_{\tau} = \frac{2000(G_c)}{\pi R^3}$
<b>Variables</b> $G_c$ = Gravitational Constant = 980.7 (cgs) or 98.07 (SI) $R$ = Radius of plates (mm) $H$ = Gap between plates (mm)	
<b>Options</b> 8, 25, 40, 50 mm sizes Serated plates Disposable plates Invar Plates	
<b>Environmental Systems</b> Ambient Oven Fluid Bath, Fluid Bath 2 * Peltier * see Chapter 2 for more details regarding lower tool	



### General Information

Parallel Plates are used to test polymer melts, soft solids, and higher viscosity fluids. Using disposable plates they are used for testing thermosetting resins and epoxy curing. The wide range of sizes, use over a wide range of viscosities, variable gap, and ease of loading, make them a very versatile tool. Additionally, high shear rates are accessible using a small gap setting.

### Operating Ranges

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using the parallel plate geometry. The following geometry-specific factors affect the operating range of parallel plate geometry:

- Plate diameter (strain constant  $K_{\gamma}$  and stress constant  $K_{\tau}$ )
- Gap between plates (strain constant  $K_{\gamma}$ )

Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor

To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type using the parallel plate geometry, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where:  $K_\tau$  = Stress Constant

$K_\gamma$  = Strain Constant

and C is computed from the following:

Transducer	C for $G^*$ MAXIMUM		C for $G^*$ MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06$ (rad/g•cm)	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 4.9 e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
2K STD 10K STD	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
100 FRT 200 FRT 100 FRTN1 200 FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	for $\omega=100$ ; $J = 2.60e-05$ for $\omega=10$ ; $J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
	$C = \frac{M_{\max}}{\theta_{\min}}$	for $\omega < 10$ ; (see note below)		

NOTE: The values for  $M$  (g•cm) and  $\theta$  (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.

To determine the maximum or minimum complex viscosity  $\eta^*$  that can be measured at a given frequency, use the following formula:

$$\eta^* = \frac{G^*}{\omega} \quad (4-2)$$



where:  $\eta^*$  = Complex viscosity (Poise)  
 $G^*$  = Complex Modulus (dynes/cm<sup>2</sup>)  
 $\omega$  = Frequency (rad/sec)

Using a spreadsheet application such as Microsoft®Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate  $G^*$  MAXIMUM and  $G^*$  MINIMUM using equation 4-1.
2. Substitute the  $G^*$  MAXIMUM value into equation 4-2, then determine the upper limit of operation by calculating  $\eta^*$  at each  $\omega$  ( $\omega$  values chosen to be from the lowest to highest frequencies within the transducer operating range).
3. Substitute the  $G^*$  MINIMUM value into equation 4-2, then determine the lower limit of operation by calculating  $\eta^*$  at each  $\omega$ .
4. Generate an X-Y scatter plot of complex viscosity  $\eta^*$  (Y axis) versus frequency  $\omega$  (X axis).

The region between the upper and lower limits of operation is the range of complex viscosity that can be tested. Appendix 1 contains tables of  $G^*$  values for some tool combinations, transducers and a standard motor.

## Tool Installation

1. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on then mount the upper and lower tools on the actuator shafts.
3. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset Torque to Zero" buttons.
4. Using the stepper control buttons, lower the stage to a point where the tools are close but not touching.
5. Use the "Zero Fixture" button in the *Set Gap/Instrument Control* function to determine the zero point for the test tools.
6. Raise the stage to provide sufficient room for sample loading.

## Sample Loading

The recommended gap setting for parallel plates is between 0.5 and 2 millimeters. Figure 4-4 shows parallel plates with sample loaded.

1. Place the sample on the lower plate. Ensure that the sample is centered on the tool.
2. Using the stepper motor buttons on the right side of the test station, adjust the sample gap until the upper plate is close to the specimen. Set the gap using the *Set Gap/Instrument Control* function, entering the appropriate parameters. If the specimen is a regularly shaped, non-flowing material, you can also manually set the gap by continuing to lower the upper plate until only a slight force is generated. The initial gap should be set approximately 0.05 mm above the final desired gap to facilitate sample trimming.
3. If the specimen is a gel or flowing material, lowering the upper plate onto the sample will result in the specimen being distributed across the lower plate into a regular cylindrical geometry. For this type of sample, using the normal force limits helps to avoid damaging the sample, as the sample may be rapidly compressed in a manual loading procedure with insufficient time for sample relaxation. In either case the initial gap should be about 0.05 mm higher than the final desired gap.
4. Trim the sample flush with the edges of the plates.

5. Lower the stage to the final gap setting. Again, this can be done manually, or using the *Set Gap/Instrument Control* function, changing the entered gap to the desired final value. The sample should bulge slightly as shown in Figure 4-4.
6. Allow samples to relax before beginning dynamic testing by monitoring the force and waiting for it to decay to close to zero.
7. Enter the sample dimension in the Orchestrator Test Geometry screen. Note that if the tools were correctly zeroed the actual sample gap can either be read from the Gap real-time parameter, or measured automatically at the start of the test by selecting the "Read Test Fixture Gap" check box in the Geometry screen.

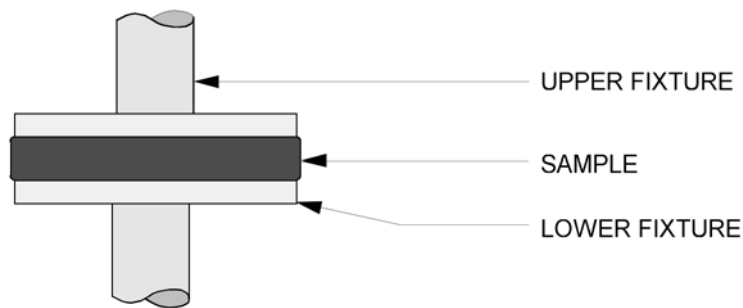
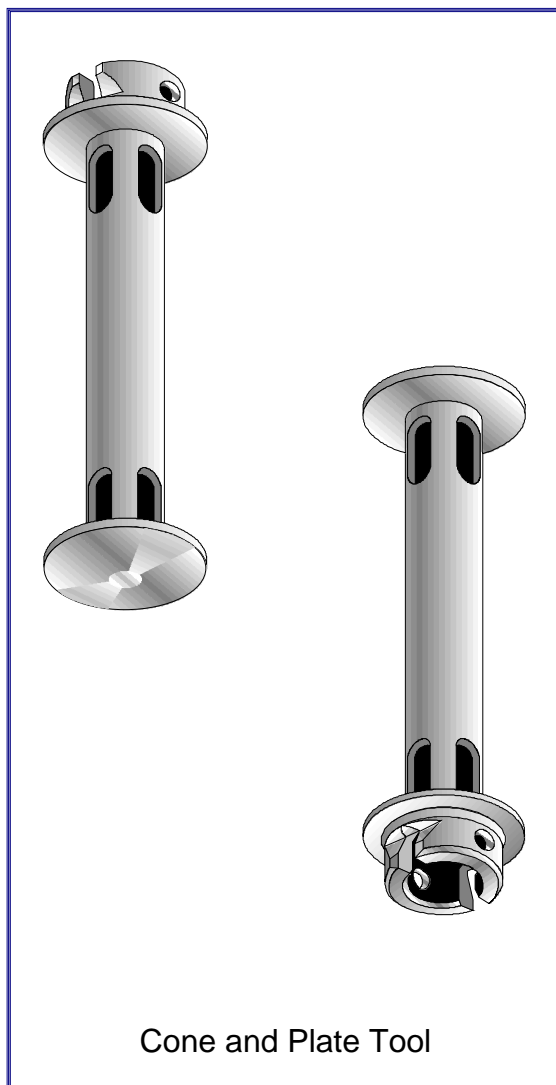


Figure 4-4. Parallel Plates with Sample Loaded

## Cone and Plate

<b>Strain Constant</b> $K_\gamma = \frac{1}{\beta}$	<b>Stress Constant</b> $K_\tau = \frac{3000(G_c)}{2\pi R^3}$
<b>Normal Stress Constant</b> $K_Z = \frac{200}{\pi R^2} * G_c$	<b>First Normal Stress Difference</b> $N_1 = K_Z * F_Z$
<b>Variables</b> $G_c$ = Gravitational constant = 980.7(cgs) or 98.07(SI) $R$ = Radius of plate (mm) $\beta$ = Cone angle (rad) $F_z$ = Normal force (g)	
<b>Options</b> 25, 50 mm Diameter; 0.02, 0.04, 0.1 rad Cone Angle Disposable cones and plates Invar cones and plates	
<b>Environmental Systems</b> Ambient Oven Fluid Bath, Fluid Bath 2 * Peltier * see Chapter 2 for more details regarding lower tool	



### General Information

The Cone and Plate tool is used in the testing of polymer melts, as well as suspensions and emulsions. By design, there is no velocity gradient across its diameter during steady shear testing. It is also used when shear normal stress measurements are desired. Unlike Parallel Plates, the gap for a Cone and Plate is fixed, and defined by the cone geometry. Because of this, the Cone and Plate is normally used for isothermal testing only, as temperature changes would lead to changes in gap due to thermal expansion. For special cases, however, Invar tools can be used. Because of Invar's exceptional thermal expansion properties, Invar tools are used for applications where temperature ramps or sweeps are necessary.

## Operating Ranges

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using the cone and plate geometry. The following geometry-specific factors affect the operating range of cone and plate geometry:

- Plate diameter (strain constant  $K_\gamma$  and stress constant  $K_\tau$ )
- Cone angle (strain constant  $K_\gamma$ )

Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor

To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type using the cone and plate geometry, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where  $K_\tau$  = Stress Constant

$K_\gamma$  = Strain Constant

and C is computed from the following:

Transducer	C for $G^*$ MAXIMUM		C for $G^*$ MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06$ (rad/g•cm)	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 4.9 e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
2K STD 10K STD	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
100 FRT 200 FRT 100 FRTN1 200 FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	for $\omega=100$ ; $J = 2.60e-05$ for $\omega=10$ ; $J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
	$C = \frac{M_{\max}}{\theta_{\min}}$	for $\omega < 10$ ; (see note below)		

NOTE: The values for  $M$  (g•cm) and  $\theta$  (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.

To determine the maximum or minimum complex viscosity  $\eta^*$  that can be measured at a given frequency, use the following formula:

$$\eta^* = \frac{G^*}{\omega} \quad (4-2)$$

where:  $\eta^*$  = Complex viscosity (Poise)  
 $G^*$  = Complex Modulus (dynes/cm<sup>2</sup>)  
 $\omega$  = Frequency (rad/sec)

Using a spreadsheet application such as Microsoft® Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate  $G^*$  MAXIMUM and  $G^*$  MINIMUM using equation 4-1.
2. Substitute the  $G^*$  MAXIMUM value into equation 4-2, then determine the upper limit of operation by calculating  $\eta^*$  at each  $\omega$  ( $\omega$  values chosen to be from the lowest to highest frequencies within the transducer operating range).
3. Substitute the  $G^*$  MINIMUM value into equation 4-2, then determine the lower limit of operation by calculating  $\eta^*$  at each  $\omega$ .
4. Generate an X-Y scatter plot of complex viscosity  $\eta^*$  (Y axis) versus frequency  $\omega$  (X axis).

The region between the upper and lower limits of operation is the range of complex viscosity that can be tested. Appendix 1 contains tables of  $G^*$  values for some tool combinations, transducers and a standard motor.

## Tool Installation

Install a tool as follows:

1. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on then mount the upper and lower tools on the actuator shafts.
3. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset torque to Zero" buttons.
4. Using the stepper control buttons, lower the stage to a point where the tools are close but not touching.
5. Use the "Zero Fixture" button in the *Set Gap/Instrument Control* function to determine the zero point for the test tools.
6. Raise the stage to provide sufficient room for sample loading.

## Gap and Cone Angle

The actual gap setting and cone angle for each cone and plate tool is stated on the *Certificate of Calibration* that is included in the tool kit. If no *Certificate of Calibration* is available, contact our Technical Service department.

## Testing at Other than Ambient Temperatures

Use the standard cone and plate tools for isothermal testing only. Transitions to elevated temperatures cause expansion of the tool. For example, with a 50-micron nominal gap setting for a 50-mm cone and plate geometry, thermal expansion can cause contact between tools, resulting in erroneous test data. The system should be equilibrated at the desired working temperature and then the final gap set.

For special applications, Invar tools can be used. Invar has a coefficient of thermal expansion that is 10 times less than steel between 0 and 230° C. Outside this range, Invar's thermal expansion properties are similar to steel's. If temperature ramps or sweeps must be performed using cone and plate tools, Invar must be used.

## Sample Loading

1. Ensure that the gap has been zeroed.
2. Place the sample on the lower plate. Ensure that the sample is centered on the tool.
3. Using the stepper motor buttons on the right side of the test station adjust the sample gap until the upper plate is close to the specimen. Set the gap using the *Set Gap/Instrument Control* function, entering the appropriate parameters. The initial gap should be set approximately 0.05 mm above the final gap to facilitate sample trimming. For softer materials, using the normal force limits helps to avoid damaging the sample as the sample may be rapidly compressed in a manual loading procedure, with insufficient time for sample relaxation.
4. Trim the sample flush with the edges of the plates.
5. Lower the stage to achieve the gap specified on the *Certificate of Calibration*. Again, this can be done manually, or using the *Set Gap/Instrument Control* function, changing the entered gap to the specified final value. The sample should bulge slightly as shown in Figure 4-5.
6. Allow samples to relax before beginning testing by monitoring the force and waiting for it to decay to close to zero.
7. Enter the sample dimension in the Orchestrator Test Geometry screen. Note that if the tools were correctly zeroed the actual sample gap can either be read from the Gap real-time parameter, or measured automatically at the start of the test by selecting the "Read Test Fixture Gap" check box in the Geometry screen.

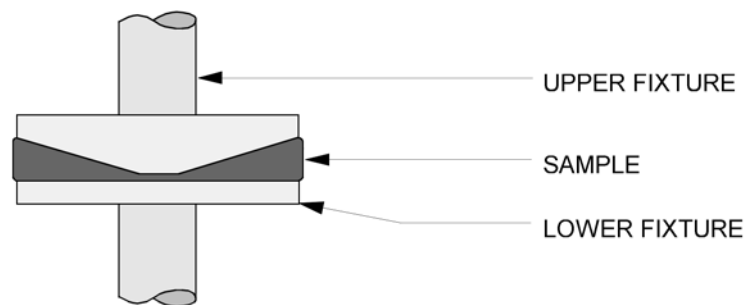
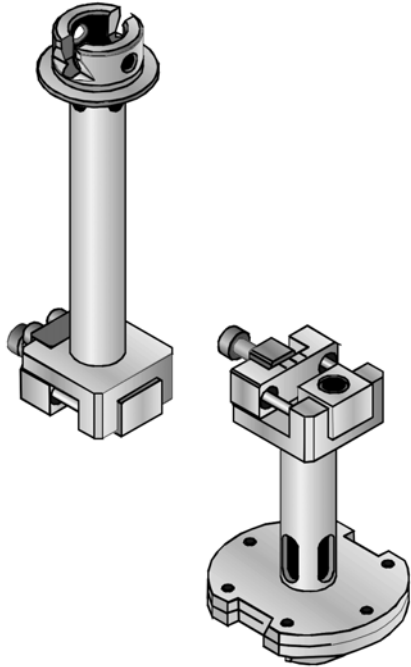


Figure 4-5. Cone and Plate with Sample Loaded

## Torsion Rectangular, New Design

<p><b>Strain Constant</b></p> $K_y = \frac{T (1/3 - 0.21T/W)(3W + 1.8T)}{W^2 L}$	<p><b>Stress Constant</b></p> $K_\tau = 1000 \left( \frac{3 + \left( 1.8 \left( \frac{T}{W} \right) \right)}{WT^2} \right) G_c$	 <p>Torsion Rectangular Tool (new design)</p>
<p><b>Variables</b></p> <p><math>G_c</math> = Gravitational constant = 980.7(cgs) or 98.07(SI)</p> <p><math>T</math> = Thickness of sample (mm)</p> <p><math>W</math> = Width of sample (mm)</p> <p><math>L</math> = Length of sample (mm)</p>		
<p><b>Options</b></p> <p>Clamps to accommodate thicknesses up to 6.5 mm</p>		
<p><b>Environmental Systems</b></p> <p>Ambient</p> <p>Oven</p>		

### General Information

The re-designed Torsion Rectangular tool is used for testing solid materials with high modulus, including thermosets, thermoplastics and elastomers. The sample is held in tension between the upper and lower tool. Three setting anvils (Figure 4-6) are provided to accommodate samples of varying thickness. Each setting anvil is designed to provide clamping for two different sample thickness ranges, which are listed on the next page.

## Sample Dimensions

To prepare samples that fit within the physical constraints of the tool, use the following guidelines:

- Maximum Sample Width: 12.7 millimeters
- Typical Sample Length: 45 millimeters
- Sample Thickness depends on the size of the setting anvil used:

SETTING ANVIL	NOMINAL THICKNESS STAMPED ON FACE	ACTUAL SAMPLE THICKNESS CLAMPING RANGE
1	0.3 mm	Up to 0.6 mm
	1.0 mm	0.6 to 1.5 mm
2	2.0 mm	1.5 to 2.5 mm
	3.0 mm	2.5 to 3.5 mm
3	4.0 mm	3.5 to 5.0 mm
	6.0 mm	5.0 to 6.5 mm

## Clamping Torque

Always use the correct size setting anvil for the sample thickness. If the sample does not fit properly in the tool, erroneous data may result. Adjusting the sliding clamps to the proper tightness is imperative. A torque screwdriver is included with this tool. For each material, some experimentation may be required to find the best clamp torque value to obtain good results. Under tightening, or for softer materials, over-tightening the clamps will result in erratic data. Once a good torque value is obtained for a specific sample (material and thickness), all subsequent samples should be tightened to the same value. Also, both clamps should always be tightened to the same torque value.

**NOTE:** Loading soft samples or samples that do not properly fit the clamps can result in inaccurate data.

## Operating Ranges

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using the torsion rectangular geometry.

The following geometry-specific factors affect the operating range of torsion rectangular geometry:

- Thickness of sample (stress constant  $K_\tau$  and strain constant  $K_\gamma$ )
- Width of sample (stress constant  $K_\tau$  and strain constant  $K_\gamma$ )
- Length of sample (strain constant  $K_\gamma$ )

Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor



To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type using the torsion rectangular geometry, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where:  $K_\tau$  = Stress Constant

$K_\gamma$  = Strain Constant

and C is computed from the following:

Transducer	C for $G^*$ MAXIMUM		C for $G^*$ MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06$ (rad/g•cm)	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	This transducer is not generally recommended for use with this tool. However, it may be used for some limited applications.			
2K STD 10K STD	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
100 FRT 200 FRT 100 FRTN1 200 FRTN1	These transducers are not recommended for use with the torsion rectangular tool.			
NOTE: The values for $M$ (g•cm) and $\theta$ (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.				

Using a spreadsheet application such as Microsoft® Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate  $G^*$  MAXIMUM and  $G^*$  MINIMUM (using equation 4-1) for a sample of fixed length and width and the minimum and maximum thickness the tool can accommodate.
2. Generate an X-Y scatter plot of sample thickness (Y axis) versus complex modulus,  $G^*$ , (X axis).

The region between the upper and lower limits of operation is the range of complex modulus that can be tested.

## Coefficient of Thermal Expansion ( $\alpha$ )

When testing at other than ambient temperatures, the coefficient of thermal expansion for Torsion Rectangular geometry is defined as:

$$\alpha = \frac{\Delta L}{\Delta t} \left( \frac{1}{L_0} \right)$$

where  $\alpha$  = Coefficient of Thermal Expansion  $\left( \frac{1}{^\circ\text{C}} \right)$

$\Delta t$  = Change in temperature ( $^\circ\text{C}$ )

$L_0$  = Original length of sample (mm)

$\Delta L$  = Change in length of sample (mm)

Positive  $\Delta L$  indicates increasing sample length

## Tool Installation

1. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on, and in dynamic mode.
3. Mount the upper and lower tools on the actuator shafts.
4. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset torque to Zero" buttons.
5. Using the stepper control buttons, lower the stage to a point where the tools are close but not touching.
6. Use the "Motor Position Offset" button in the *Set Gap/Instrument Control* function to ensure that the upper and lower tool openings are aligned.
7. For 2K FRT transducers only (skip this for STD transducers), use the "Zero Fixture" button in the *Set Gap/Instrument Control* function to bring the tools together and determine the zero point for the test tools.
8. Raise the stage to provide sufficient room for sample loading.

## Sample Loading

Refer to Figure 4-6 and Figure 4-7 during the following procedure.



**WARNING:** This is a high-torque motor. Turning on the motor while in dynamic mode causes the motor to snap to dynamic zero position at a high velocity. This can cause severe damage to the transducer and/or personal injury. To avoid damaging yourself and the transducer:

**Never turn on the motor while a sample is loaded.**

**Keep hands clear of the motor.**

---

1. Measure and record the following sample dimensions:
  - Width
  - Thickness
  - Length (STD transducers only - length will be determined from gap setting for 2K FRT transducers)
2. Select a matching pair of setting anvils (based upon sample thickness) and secure them in the upper and lower tool. Please note that each setting anvil is machined in such a way as to provide mounting for two different sample thickness ranges. A "nominal" thickness (roughly the center of the clamping range) is stamped on two of the anvil faces (opposite sides). The setting anvil should be mounted such that the desired "nominal" thickness is visible from the outside "back" of the tool (opposite the actual sample).
3. Place the sample into the lower tool. Center the sample in the tool using the reference lines scribed in the setting anvil and sliding clamp. Partially tighten the clamp (using the adjusting screw) to hold the sample.
4. Lower the stage until the upper tool is about 1/4-inch from the sample.
5. Use the "Motor Position Offset" button in the *Set Gap/Instrument Control* function in Orchestrator, to radially align the sample with the upper tool if necessary.



**CAUTION:** In the next step, do not generate a Torque or Normal Force greater than 50% of full-scale. Failure to observe this caution may result in damage to the transducer.

---

6. While confirming the sample fits into the upper tool, lower the stage until a compressive (downward) Normal Force of about 10% of full-scale is generated. If the sample is not aligned properly, re-raise the stage and carefully realign the sample and tool using the "Motor Position Offset" button.
7. Ensure (visually) that the sample is completely inserted into the tools.
8. Tighten the lower and upper sliding clamps (using the adjusting screw) to the desired torque using the torque screwdriver.
9. Raise the stage until a force of approximately 10% of full-scale is generated. Please note that this tension level is a general recommendation only, and you should set the tension level according to the sample characteristics, with thinner and/or lower modulus materials requiring less axial force. When using the AutoTension feature, adjust the stage so that the normal force is zero.

10. Using the "Motor Position Offset" slider in the *Set Gap/Instrument Control* function in Orchestrator adjust the motor position until the displayed torque is zero.  
**DO NOT** use the "Offset torque to Zero" button.
11. Read the gap and record this distance as the sample length (2K FRT transducers only).
12. Use the **Hold** function under the Control pull down menu when changing temperature.

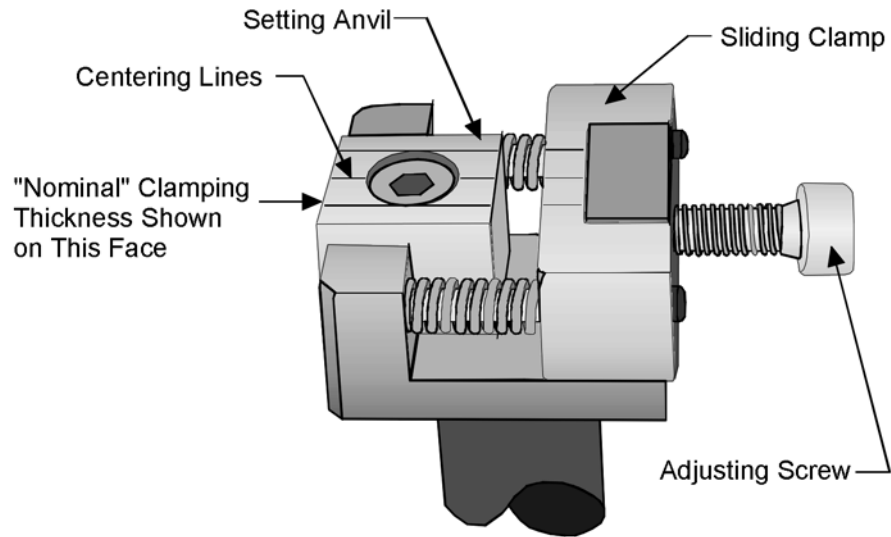


Figure 4-6. New Torsion Rectangular Tool Details

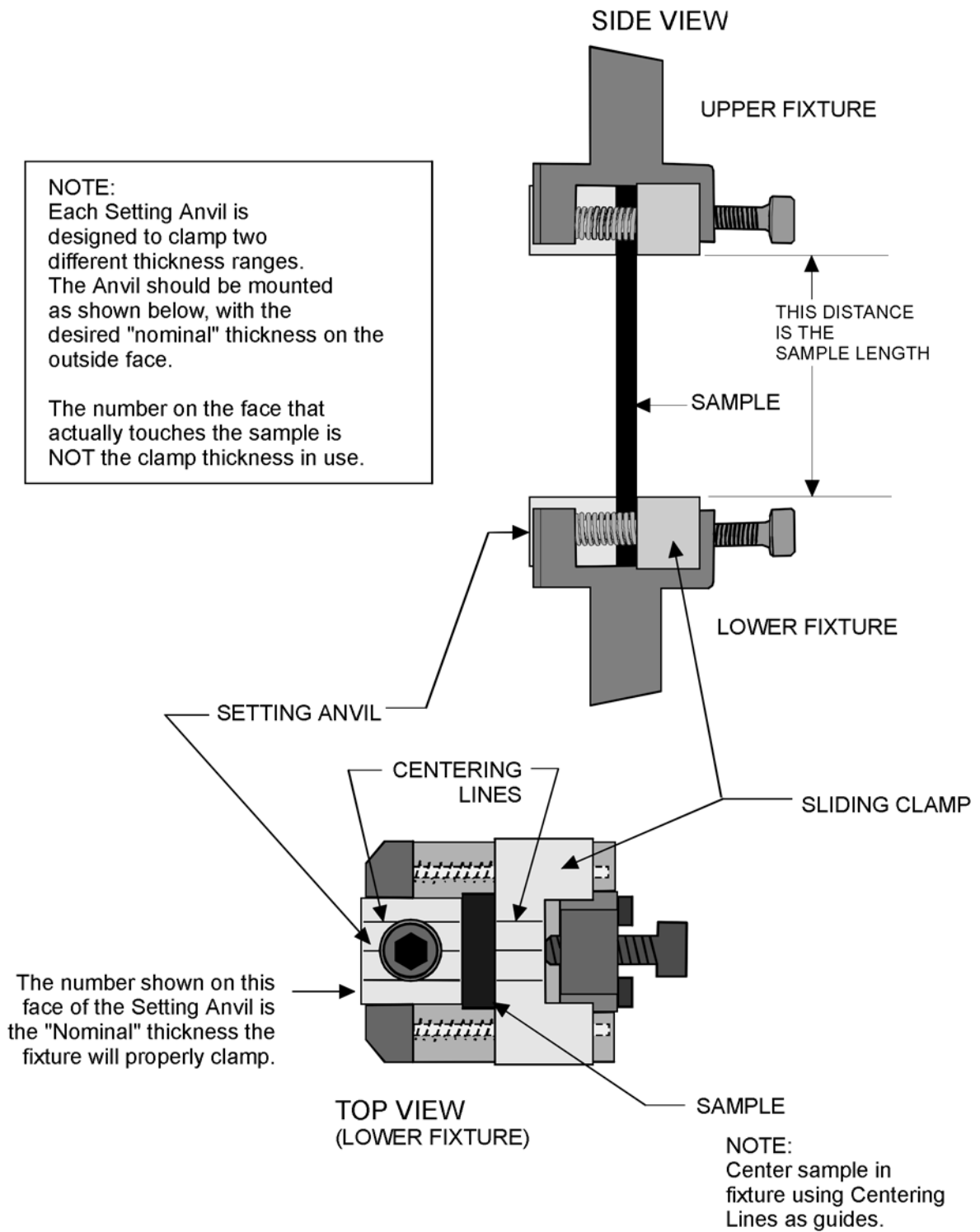
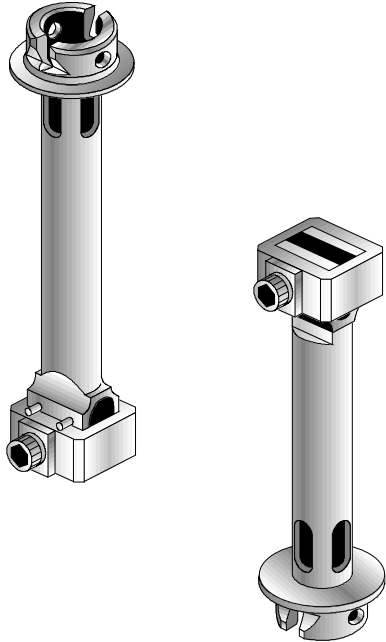


Figure 4-7. Torsion Rectangular with Sample Loaded

NOTE: For more information regarding test setup and usage of the Orchestrator software program, please refer to the Orchestrator Getting Started Guide or to the online help available within the program.

## Torsion Rectangular, Original Design

<p><b>Strain Constant</b></p> $K_{\gamma} = \frac{T}{L} \left( 1 - \left( 0.378 \left( \frac{T}{W} \right)^2 \right) \right)$	<p><b>Stress Constant</b></p> $K_{\tau} = 1000 \left( \frac{3 + \left( 1.8 \left( \frac{T}{W} \right) \right)}{WT^2} \right) G_c$	 <p>Torsion Rectangular Tool (original design)</p>
<p><b>Variables</b></p> <p><math>G_c</math> = Gravitational constant = 980.7(cgs) or 98.07(SI)</p> <p><math>T</math> = Thickness of sample (mm)</p> <p><math>W</math> = Width of sample (mm)</p> <p><math>L</math> = Length of sample (mm)</p>		
<p><b>Options</b></p> <p>Insets to accommodate thicknesses from 0.76 mm to 6.35 mm</p>		
<p><b>Environmental Systems</b></p> <p>Ambient</p> <p>Oven</p>		

### General Information

The Torsion Rectangular tool is used for testing solid materials with high modulus, including thermosets, thermoplastics and elastomers. The sample is held in tension between the upper and lower tool. Several inserts are provided to accommodate samples of varying thicknesses.

### Sample Dimensions

To prepare samples that fit within the physical constraints of the tool, use the following guidelines:

- Maximum Sample Width: 12.7 millimeters
- Typical Sample Length: 45 millimeters
- Sample Thickness depends on the size of the insert used:

INSERT DESIGNATION	MAXIMUM SAMPLE THICKNESS (MM)
Insert 1	0.85
Insert 2	1.62
Insert 3	3.15

Always use the correct size insert for the sample thickness. If the sample does not fit tightly in the insert, erroneous data may result. Shims can be used for very thin samples, with the stipulation that an identical number of equally-sized shims must be used on either side of the sample.

**NOTE:** Loading soft samples or samples that do not properly fit the inserts can result in inaccurate data.

## Operating Ranges

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using the torsion rectangular geometry.

The following geometry-specific factors affect the operating range of torsion rectangular geometry:

- Thickness of sample (stress constant  $K_\tau$  and strain constant  $K_\gamma$ )
- Width of sample (stress constant  $K_\tau$  and strain constant  $K_\gamma$ )
- Length of sample (strain constant  $K_\gamma$ )

Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor

To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type using the torsion rectangular geometry, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where  $K_\tau$  = Stress Constant

$K_\gamma$  = Strain Constant

and C is computed from the following:



Transducer	C for G* MAXIMUM		C for G* MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06 \text{ (rad/g}\cdot\text{cm)}$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	This transducer is not generally recommended for use with this tool. However, it may be used for some limited applications.			
2K STD 10K STD	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
100 FRT 200 FRT 100 FRTN1 200 FRTN1	These transducers are not recommended for use with the torsion rectangular tool.			
NOTE: The values for $M$ (g•cm) and $\theta$ (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.				

Using a spreadsheet application such as Microsoft® Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate G\* MAXIMUM and G\* MINIMUM (using equation 4-1) for a sample of fixed length and width and the minimum and maximum thicknesses the tool can accommodate.
2. Generate an X-Y scatter plot of sample thickness (Y axis) versus complex modulus,  $G^*$ , (X axis).

The region between the upper and lower limits of operation is the range of complex modulus that can be tested.

## Coefficient of Thermal Expansion ( $\alpha$ )

When testing at other than ambient temperatures, the coefficient of thermal expansion for Torsion Rectangular geometry is defined as:

$$\alpha = \frac{\Delta L}{\Delta t} \left( \frac{1}{L_0} \right)$$

where  $\alpha$  = Coefficient of Thermal Expansion  $\left( \frac{1}{^\circ\text{C}} \right)$

$\Delta t$  = Change in temperature ( $^\circ\text{C}$ )

$L_0$  = Original length of sample (mm)

$\Delta L$  = Change in length of sample (mm)

Positive  $\Delta L$  indicates increasing sample length

## Tool Installation

1. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on, and in dynamic mode.
3. Mount the upper and lower tools on the actuator shafts.
4. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset torque to Zero" buttons.
5. Using the stepper control buttons, lower the stage to a point where the tools are close but not touching.
6. Use the "Motor Position Offset" button in the *Set Gap/Instrument Control* function to ensure that the upper and lower tool openings are aligned.
7. For 2K FRT transducers only (skip this for STD transducers) use the "Zero Fixture" button in the *Set Gap/Instrument Control* function to bring the tools together and determine the zero point for the test tools.
8. Raise the stage to provide sufficient room for sample loading.

## Sample Loading

This procedure is for use with 2K FRT and STD transducers **only**. Refer to Figure 4-8 during the following procedure.



### **WARNING:**

#### **POSSIBLE PERSONAL INJURY**

#### **POSSIBLE DAMAGE TO INSTRUMENT**

**This is a high-torque motor. Turning on the motor while in dynamic mode causes the motor to snap to dynamic zero position at a high velocity. This can cause severe damage to the transducer and/or personal injury. To avoid damaging yourself and the transducer:**

**Never turn on the motor while a sample is loaded.**

**Keep hands clear of the motor.**

---

1. Measure and record the following sample dimensions:
  - Width
  - Thickness
  - Length (STD transducers only - length will be determined from gap setting for 2K FRT transducers)
2. Select an insert and place the sample between the inserts.
3. Place the sample (with inserts) into the lower tool then place both collars (one above the other) over the sample. Ensure that the lower collar rests flush against the four stop pins in the lower tool.
4. Lower the stage until the upper tool is about 1/4-inch from the sample.
5. Open the *Set Gap/Instrument Control* function. Use the "Motor Position Offset" button to radially align the sample with the upper tool if necessary.



---

**CAUTION:** In the next step, do not generate a Torque or Normal Force greater than 50% of full-scale. Failure to observe this caution may result in damage to the transducer.

---

6. While confirming the upper insert (and sample) fits into the upper tool, lower the stage until a compressive (downward) Normal Force of about 10% of full-scale is generated. If the sample is not aligned properly, re-raise the stage and realign the sample and tool using the "Motor Position Offset" button.
7. Ensure (visually) that the insert is resting on the four small pins that are located directly below the lower (and above the upper) insert.
8. Slide the upper collar up onto the upper tool, ensuring that the collar rests flush against the four stop pins in the tool. Tighten the collar just enough to hold it in place.
9. Tighten the lower and upper collars.
10. Raise the stage until a force of approximately 10% of full-scale is generated. Please note that this tension level is a general recommendation only, and you should set the tension level according to the sample characteristics. When using the AutoTension feature, adjust the stage so that the normal force is zero.
11. Using the "Motor Position Offset" slider in the *Set Gap/Instrument Control* function in Orchestrator adjust the motor position until the displayed torque is zero.  
**DO NOT** use the "Offset torque to Zero" button.
12. Read the gap and record this distance as the sample length (2K FRT transducers only).
13. Use **Hold** function under the Control pull down menu when changing temperature.

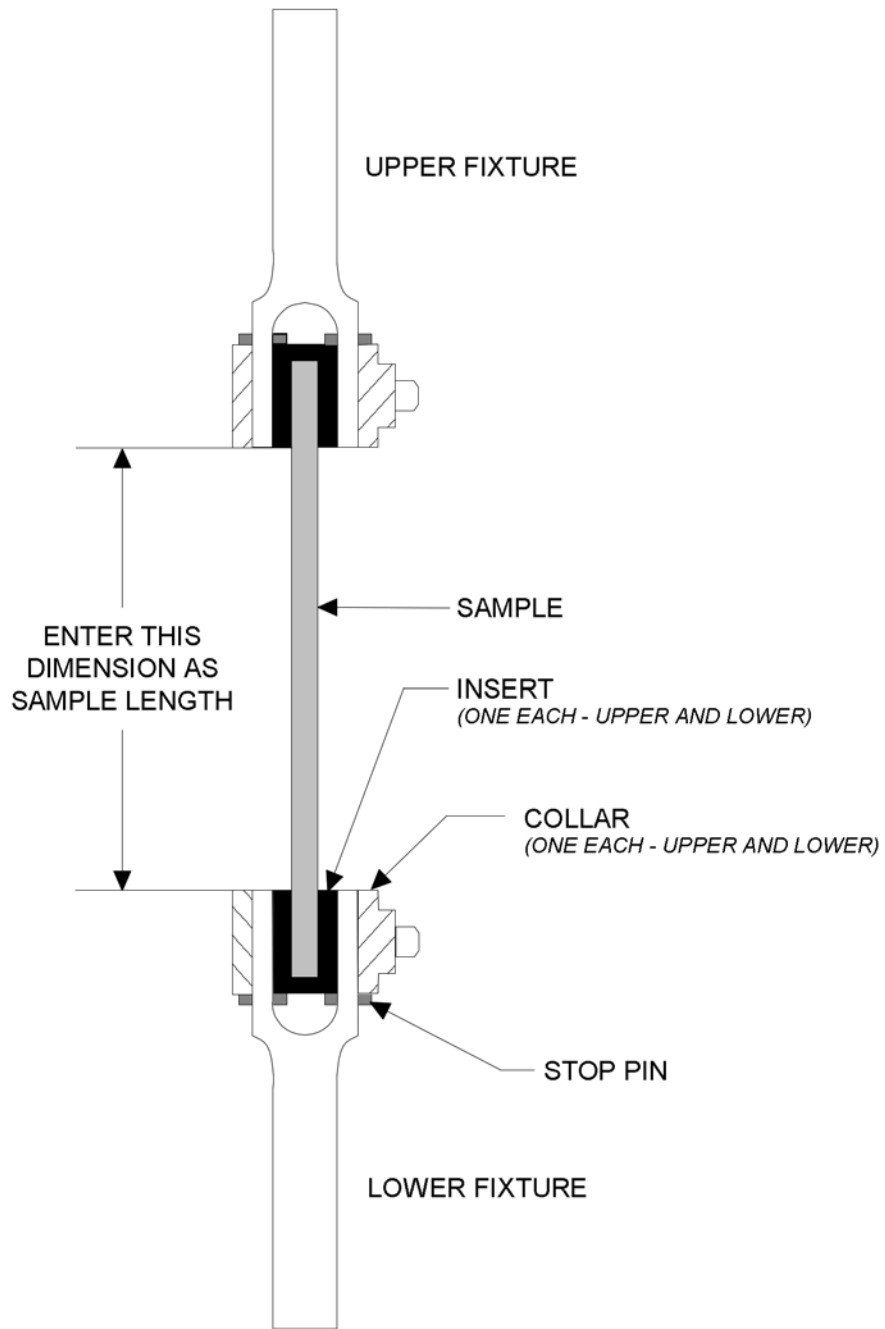
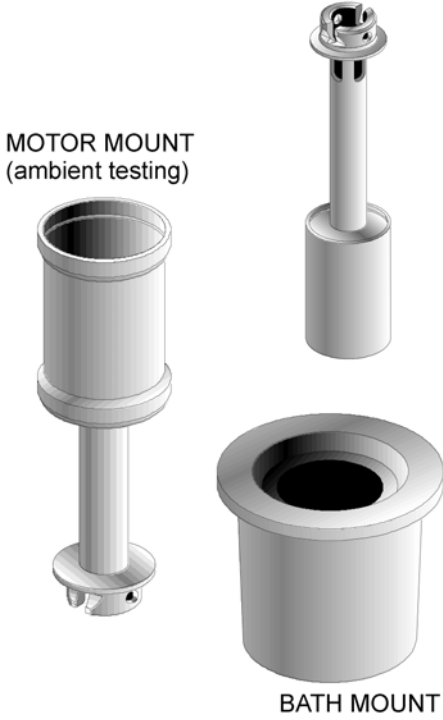


Figure 4-8. Torsion Rectangular with Sample Loaded

## Couette

<p><b>Strain Constant</b></p> $K_\gamma = \frac{2}{1 - \left(\frac{R_B}{R_C}\right)^2}$	<p><b>Stress Constant</b></p> $K_\tau = \frac{(1000)(G_c)}{2\pi L (R_B)^2}$	 <p>MOTOR MOUNT (ambient testing)</p> <p>BATH MOUNT</p> <p>Couette Tool</p>
<p><b>Variables</b></p> <p><math>G_c</math> = Gravitational constant = 980.7(cgs) or 98.07(SI)</p> <p><math>L</math> = Length of bob (mm)</p> <p><math>R_B</math> = Radius of bob (mm)</p> <p><math>R_C</math> = Radius of cup (mm)</p>		
<p><b>Options</b></p> <p>25 mm bob / 27 mm cup (ambient testing only)</p> <p>32 mm bob / 34mm cup (for fluid bath or fluid bath 2)</p> <p>16.5 mm bob / 17mm cup (for fluid bath or fluid bath 2)</p>		
<p><b>Enviromental Systems</b></p> <p>Ambient</p> <p>Oven</p> <p>Fluid Bath, Fluid Bath 2 *</p> <p>* see Chapter 2 for more details regarding lower tool</p>		

### General Information

The Couette tool is used for testing lower viscosity fluids that would not generate enough torque using parallel plates. It is also used where containment of the fluid would be difficult using other tools.

### Operating Ranges

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using the Couette. The following geometry-specific factors affect the operating range of the Couette:

- Length of bob (stress constant  $K_\tau$ )
- Radius of bob (strain constant  $K_\gamma$  and stress constant  $K_\tau$ )
- Radius of cup (strain constant  $K_\gamma$ )

Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor

To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type using the Couette, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where  $K_\tau$  = Stress Constant

$K_\gamma$  = Strain Constant

and C is computed from the following:

Transducer	C for $G^*$ MAXIMUM		C for $G^*$ MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06$ (rad/g•cm)	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 4.9 e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
2K STD 10K STD	These transducers are not recommended for use with the Couette.			
100 FRT 200 FRT 100 FRTN1 200 FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	for $\omega=100$ ; $J = 2.60e-05$ for $\omega=10$ ; $J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
	$C = \frac{M_{\max}}{\theta_{\min}}$	for $\omega < 10$ ; (see note below)		
NOTE: The values for $M$ (g•cm) and $\theta$ (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.				

To determine the maximum or minimum complex viscosity  $\eta^*$  that can be measured at a given frequency, use the following formula:

$$\eta^* = \frac{G^*}{\omega} \quad (4-2)$$

where  $\eta^*$  = Complex viscosity (Poise)  
 $G^*$  = Complex Modulus (dynes/cm<sup>2</sup>)  
 $\omega$  = Frequency (rad/sec)

Using a spreadsheet application such as Microsoft® Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate  $G^*$  MAXIMUM and  $G^*$  MINIMUM using equation 4-1.
2. Substitute the  $G^*$  MAXIMUM value into equation 4-2, then determine the upper limit of operation by calculating  $\eta^*$  at each  $\omega$  ( $\omega$  values chosen to be from the lowest to highest frequencies within the transducer operating range).
3. Substitute the  $G^*$  MINIMUM value into equation 4-2, then determine the lower limit of operation by calculating  $\eta^*$  at each  $\omega$ .
4. Generate an X-Y scatter plot of complex viscosity  $\eta^*$  (Y axis) versus frequency  $\omega$  (X axis).

The region between the upper and lower limits of operation is the range of complex viscosity that can be tested. Appendix 1 contains tables of  $G^*$  values for some combinations of bobs and cups using various transducers and a standard motor.

## Tool Installation

If you are installing the lower tool into either of the fluid baths, please refer to the appropriate section of Chapter 2 (for your specific bath) for additional information before installing the tool into the bath.

1. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on then mount the upper tools on the transducer shaft and lower tool into the fluid bath or actuator shaft depending upon which environmental control system is being used.
3. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset Torque to Zero" buttons.

## Sample Loading

In general, pour the sample into the cup then lower the bob until the upper surface of the bob is 1 to 2 mm below the surface of the sample (Figure 4-9). Note that the upper surface of the bob must be between zero and five millimeters below the upper surface of the cup. If this is not the case, adjust the sample volume accordingly. Nominal sample volumes are as follows:

Bob / Cup size	Sample Volume
16.5 mm / 17 mm	4 ml
25 mm / 27 mm	8 ml
32 mm / 34 mm	15 ml
Vane / 34 mm	40 ml

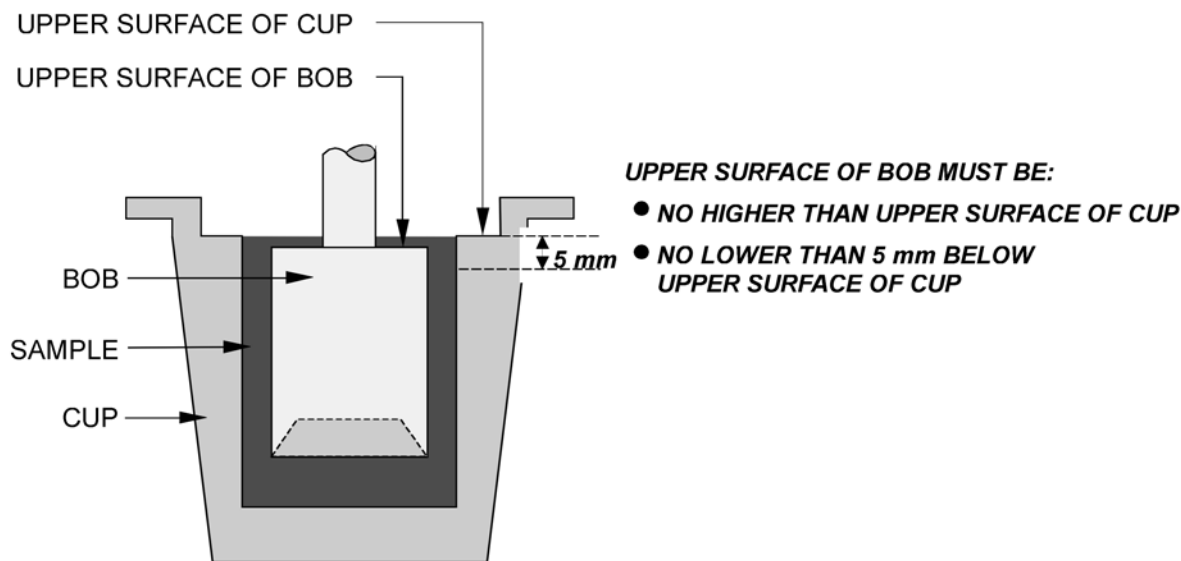


Figure 4-9. Couette with Sample Loaded

## Vane Tool

The Vane Tool is designed to replace the bob under certain circumstances. It is primarily used with materials that are highly structured, such as foams and lotions, which may tend to slip with a normal bob. This slippage could mistakenly be interpreted as a yield stress using a normal bob. For these fluids, when using the vane tool, the material within the vanes moves as a solid plug. However, for less shear thinning fluids, there will be secondary flows between the vanes, which will result in incorrect viscosity shear rate data, so it is important to use the correct tool for the material. The vane tool is also useful when testing "chunky" fluids, or that contain larger particles. Many food items would fall into this category.

The vane tool is used to do Creep/Recovery (to measure Yield Stress) or low speed Steady shear testing. The Vane Tool should only be used for low speed steady testing.

The vane tool is handled similarly to the standard bob. The Couette Geometry should still be selected in Orchestrator. The tool dimensions are determined as shown in Figure 4-10. The vane tool is designed for use with the 34-mm cup. The vane tool should be lowered carefully into the sample to minimize disturbing the sample.



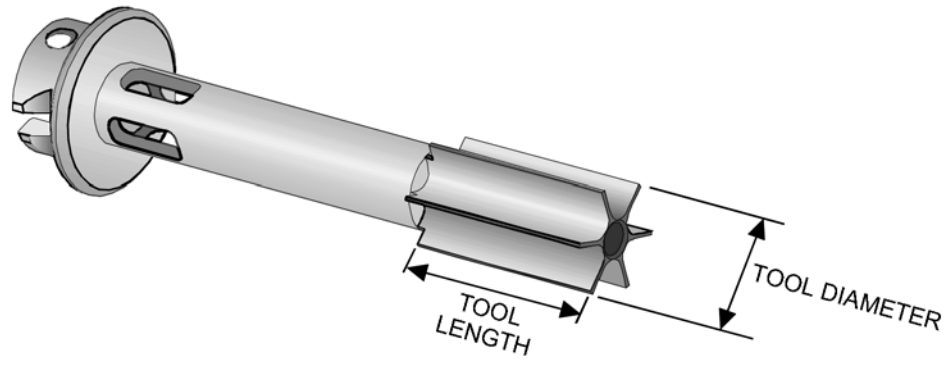
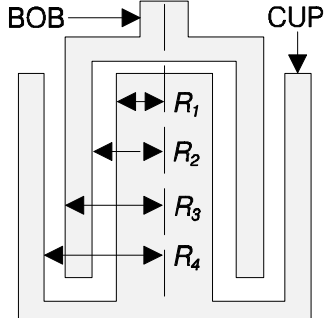
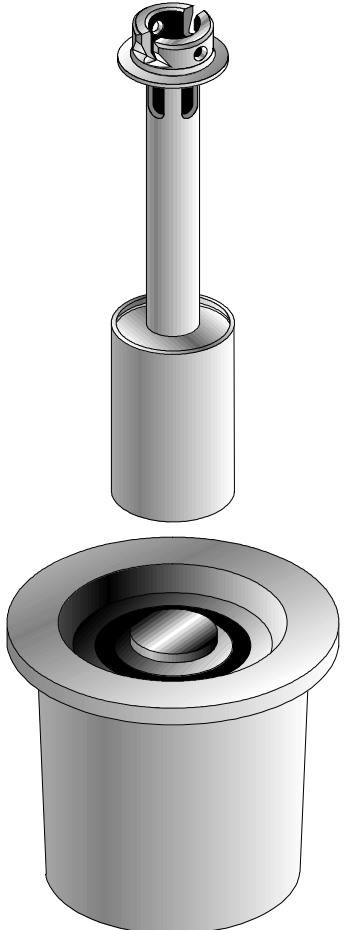


Figure 4-10. Vane Tool

## Double Wall Couette

<p><b>Strain Constant</b></p> $K_\gamma = \frac{1}{\left(\frac{R_2}{R_1}\right)^2 - 1} + \frac{1}{1 - \left(\frac{R_3}{R_4}\right)^2}$	<p><b>Stress Constant</b></p> $K_\tau = \frac{(1000)(G_c)}{2\pi L \left( (R_2)^2 + (R_3)^2 \right)}$				
<p><b>Variables</b></p> <p><math>G_c</math> = Gravitational constant = 980.7(cgs) or 98.07(SI)</p> <p>L = Length of bob (mm)</p> <p>R1 R2 R3 R4 equal the following radii:</p> 					
<p><b>Options</b></p> <table border="0"> <tr> <td>CUP</td> <td>BOB</td> </tr> <tr> <td>34 mm OD / 27.95 ID</td> <td>32 mm OD / 29.5 ID</td> </tr> </table>		CUP	BOB	34 mm OD / 27.95 ID	32 mm OD / 29.5 ID
CUP	BOB				
34 mm OD / 27.95 ID	32 mm OD / 29.5 ID				
<p><b>Environmental Systems</b></p> <p>Fluid Bath, Fluid Bath 2 *</p> <p>* see Chapter 2 for more details regarding lower tool</p>					



**Double Wall Couette Tool**

### General Information

The double wall Couette tool is used for testing lower viscosity fluids that would not generate enough torque using parallel plates. It is also used where small sample volumes are necessary.

## Operating Ranges

Operating range is defined as the region bounded by the maximum and minimum complex modulus  $G^*$  that can be measured by each transducer type using the double wall Couette geometry. The following geometry-specific factors affect the operating range of the double wall Couette:

- Inner diameter of cup,  $R_1$  (strain constant  $K_\gamma$ )
- Outer diameter of cup,  $R_4$  (strain constant  $K_\gamma$ )
- Inner diameter of bob,  $R_2$  (stress constant  $K_\tau$  and strain constant  $K_\gamma$ )
- Outer diameter of bob,  $R_3$  (stress constant  $K_\tau$  and strain constant  $K_\gamma$ )
- Length of bob (stress constant  $K_\tau$ )

Additionally, the following instrument-specific factors affect the operating range of all geometries:

- Minimum torque that can be measured by the transducer
- Transducer compliance
- Maximum strain that can be generated by the motor

To calculate the minimum and maximum  $G^*$  that can be measured by each transducer type using the double wall Couette, use the following equation:

$$G^* = \left( \frac{K_\tau}{K_\gamma} \right) C \quad (4-1)$$

where  $K_\tau$  = Stress Constant

$K_\gamma$  = Strain Constant

and C is computed from the following:

Transducer	C for $G^*$ MAXIMUM		C for $G^*$ MINIMUM	
	Equation	Values	Equation	Values
2K FRTN1 2K FRTN1E	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 1.15e-06$ (rad/g•cm)	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
1K FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	$J = 4.9 e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
2K STD 10K STD	These transducers are not recommended for use with the Double Wall Couette.			
100 FRT 200 FRT 100 FRTN1 200 FRTN1	$C = 0.1 \left( \frac{1}{J} \right)$	for $\omega=100$ ; $J = 2.60e-05$ for $\omega=10$ ; $J = 2.60e-06$	$C = \frac{M_{\min}}{\theta_{\max}}$	(see note below)
	$C = \frac{M_{\max}}{\theta_{\min}}$	for $\omega < 10$ ; (see note below)		
NOTE: The values for $M$ (g•cm) and $\theta$ (rad) are found in the specification tables in Chapter 1 (Table 1-6 through Table 1-13). Pick the correct values for your specific transducer and motor combination.				

To determine the maximum or minimum complex viscosity  $\eta^*$  that can be measured at a given frequency, use the following formula:

$$\eta^* = \frac{G^*}{\omega} \quad (4-2)$$

where  $\eta^*$  = Complex viscosity (Poise)  
 $G^*$  = Complex Modulus (dynes/cm<sup>2</sup>)  
 $\omega$  = Frequency (rad/sec)

Using a spreadsheet application such as Microsoft® Excel™, you can use the equations above to plot the range of complex viscosity that can be tested for a given geometry/transducer combination as follows:

1. Calculate  $G^*$  MAXIMUM and  $G^*$  MINIMUM using equation 4-1.
2. Substitute the  $G^*$  MAXIMUM value into equation 4-2, then determine the upper limit of operation by calculating  $\eta^*$  at each  $\omega$  ( $\omega$  values chosen to be from the lowest to highest frequencies within the transducer operating range).
3. Substitute the  $G^*$  MINIMUM value into equation 4-2, then determine the lower limit of operation by calculating  $\eta^*$  at each  $\omega$ .
4. Generate an X-Y scatter plot of complex viscosity  $\eta^*$  (Y axis) versus frequency  $\omega$  (X axis).

The region between the upper and lower limits of operation is the range of complex viscosity that can be tested. Appendix 1 contains a table of  $G^*$  values for the double wall Couette for some transducers and a standard motor.

**NOTE:** The cup installation and use procedures are different for the original fluid bath and the fluid bath 2. Please ensure that you identify which bath and Couette cup you have, and follow the correct procedures for your specific bath/cup arrangement.

## Tool Installation, Original Fluid Bath

The Double Wall Couette lower tool (cup) mounts into the fluid bath or fluid bath 2 (see next section) to ensure precise thermal control. Install the bath on the test station prior to mounting the tool in the bath.



**CAUTION:** Never place any lower tool into the bath if the temperature of the lower tool is cooler than that of the bath. Placing a tool into a warmer bath will result in expansion of the tool during use. After expansion, the tool may not be removable without damaging your bath.

We suggest that you partially insert the tool by placing a phenolic spacer between the upper lip of the lower tool, and the surface of the bath. Allow the lower tool temperature to match that of the bath, then remove the spacer and fully insert the lower tool.

1. Assemble the lower tool (cup) by installing the inner cup into the outer cup using the mounting screw provided (Figure 4-12). Make sure the o-ring is present, clean, and free of defects.
2. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
3. Verify that the motor is on then mount the upper tool on the transducer shaft and lower tool and PRT into the properly installed fluid bath.
4. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset Torque to Zero" buttons.
5. Using the manual stage control buttons, lower the stage until the upper surface of the bob is below the upper surface of the cup (Figure 4-11A).
6. Place a straight edge across the upper surface of the cup. While monitoring the normal force, raise the stage slowly until the upper surface of the bob touches the straight edge. There will be a visible increase in normal force when the bob contacts the straight edge (Figure 4-11B).
7. Zero the gap using the "Zero Indicator" button in the *Set Gap/Instrument Control* function.
8. Raise the stage to provide sufficient room for sample loading.

## Sample Loading, Original Fluid Bath

The nominal sample volume is 8 ml. In general, pour the sample into the cup, then lower the bob until the gap again reads zero, and the sample appears as shown in Figure 4-11C. If the gap is set as described in the previous section this will ensure that the nominal bob length is 31.90 mm. Note that the upper surface of the bob must be as flush as possible to the upper surface of the cup.

## Tool Maintenance

To facilitate cleaning of the lower tool, the inner cup can be removed from the outer cup when necessary. Inspect the o-ring for cuts or other damage and replace it if necessary.

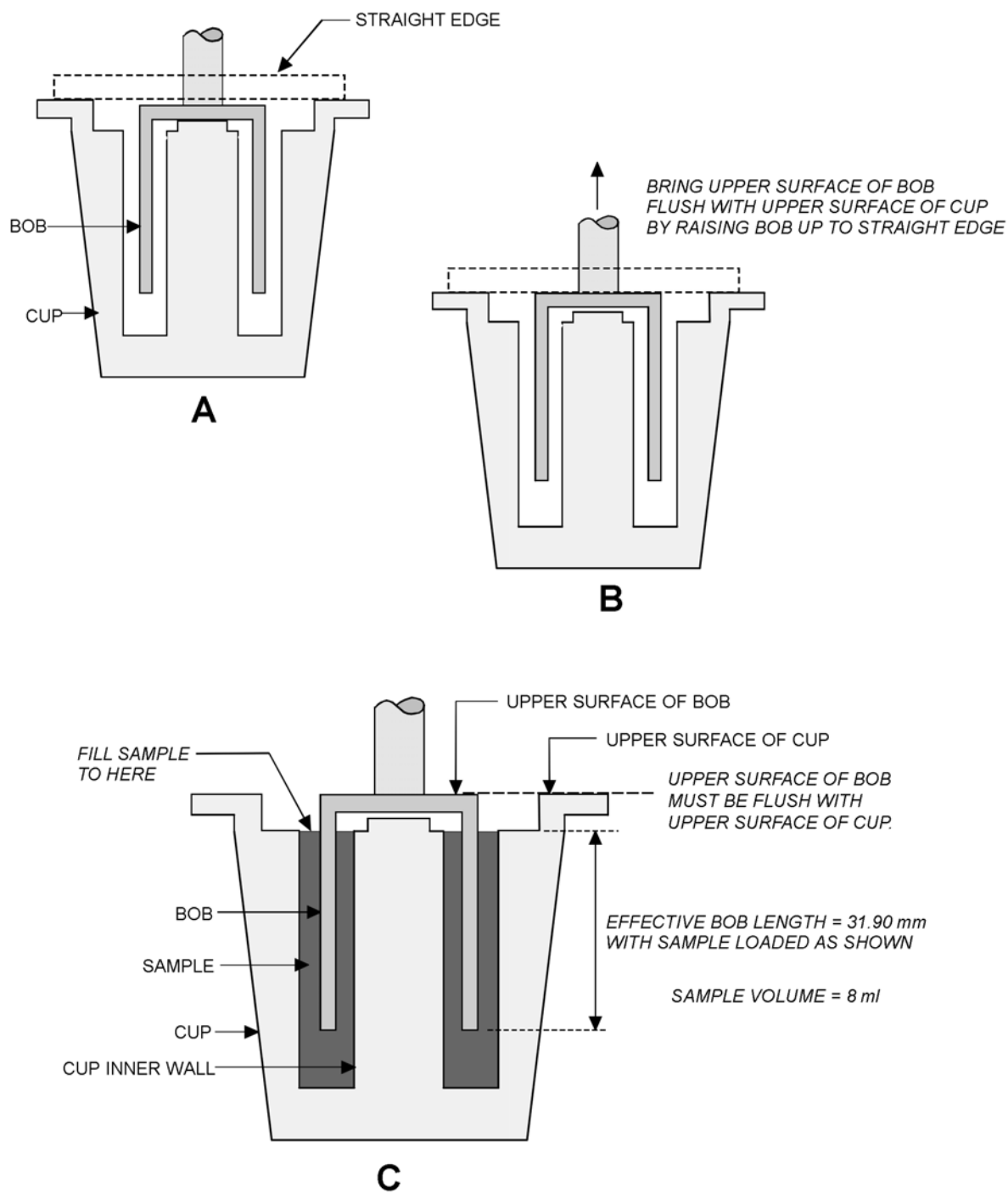


Figure 4-11. Double Wall Couette for the Original Fluid Bath - Set Up and Use

A and B - Setting the Gap, C - With Sample Loaded.

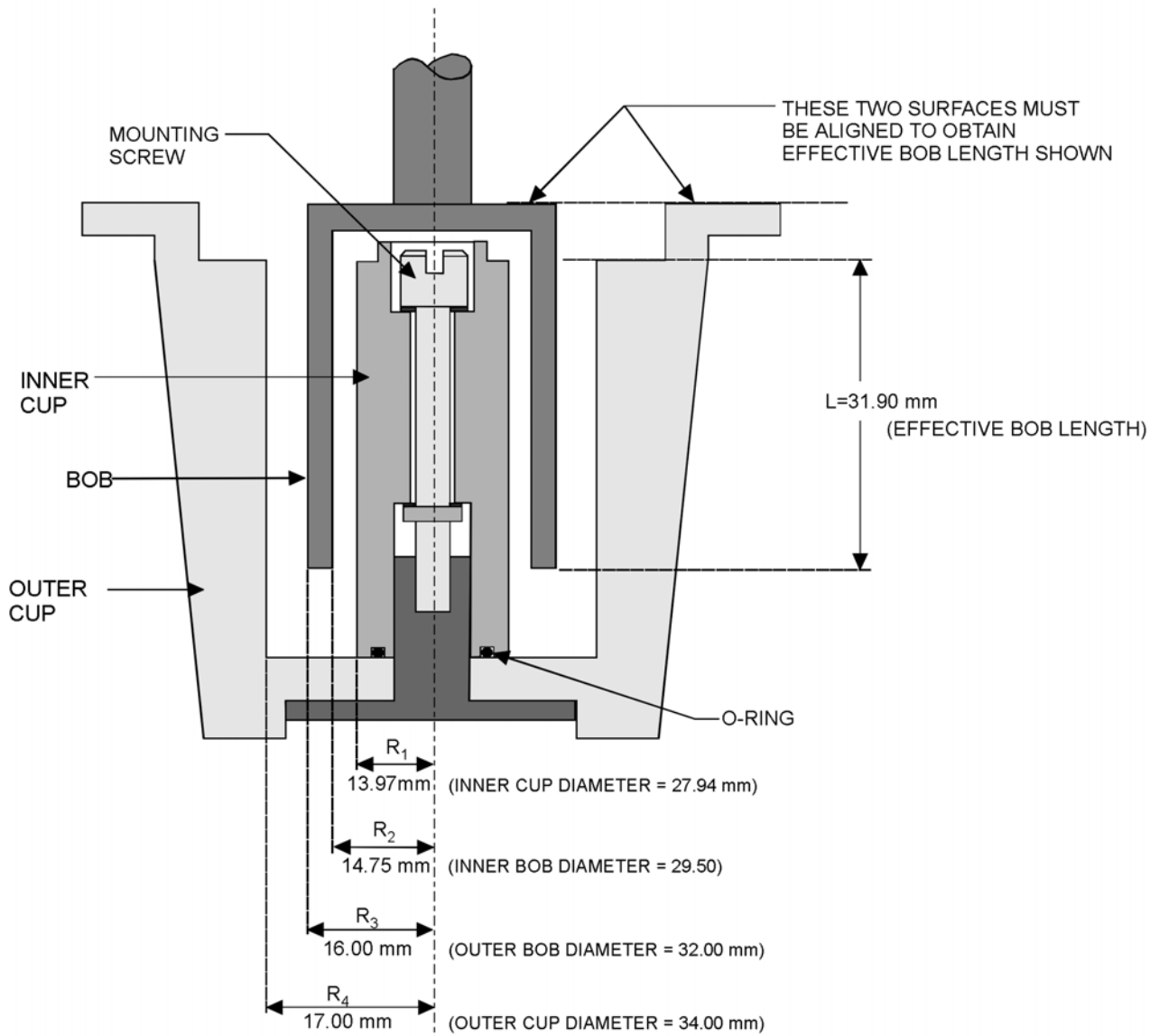


Figure 4-12. Double Wall Couette

(Original fluid bath set up showing inner and outer cup as well as tool dimensions.)

## Tool Installation, Fluid Bath 2

The Double Wall Couette lower tool (cup) mounts into the Fluid Bath 2 (see Chapter 2) to ensure precise thermal control. Install the bath on the test station prior to mounting the tool in the bath.



**CAUTION:** Never place any lower tool into the bath if the temperature of the lower tool is cooler than that of the bath. Placing a tool into a warmer bath will result in expansion of the tool during use. After expansion, the tool may not be removable without damaging your bath.

**We suggest that you partially insert the tool by placing the tool loosely into the bath. Allow the lower tool temperature to match that of the bath, then fully thread the lower tool into the bath.**

1. Select the *Set Gap/Instrument Control* function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on, then mount PRT and lower tool (Cup) into the Fluid Bath 2, and the upper tool (Bob) on the transducer shaft.
3. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset Torque to Zero" buttons.
4. Zero the gap using the "Zero Indicator" button in the *Set Gap/Instrument Control* function. Using a "Maximum Allowed Force" of 100 gm works well.
5. Using the *Set Gap/Instrument Control* function, set the Gap to 1 mm.
6. Raise the stage to provide sufficient room for sample loading.

## Sample Loading, Fluid Bath 2

The nominal sample volume is between 8 to 9 ml. For lower viscosity fluids, a volume closer to 9 ml is optimal. For higher viscosity fluids, using a volume closer to 8 ml gives good results. In all cases, the fluid level must be at least up to the sample fill level "lip". To avoid edge and boundary layer errors, filling slightly past the "lip" is desirable as shown in Figure 4-14. Overfilling the sample however, especially in the case of higher viscosity fluids, may result in errors due to an actual wetted bob length longer than the entered effective length.

Carefully pour the sample into the cup, making sure not to spill sample into the recess that holds the inner cup mounting screw. A small graduated cylinder can be used to transfer material into the Couette, as well as a syringe or pipette. In any case, a consistent sample volume from sample to sample is desirable for each material tested.

After filling the cup, return the bob back to the 1 mm gap. This will ensure that the nominal bob length is the correct 32 mm.



## Tool Maintenance, Fluid Bath 2

To facilitate cleaning of the lower tool, the inner cup can be removed from the outer cup. Unscrew the inner cup mounting screw and remove the inner cup and O-ring (Figure 4-13). Clean the cup as necessary. Reassemble the lower tool (cup) by installing the inner cup into the outer cup using the inner cup mounting screw. Before reassembling the tool, inspect the O-ring for cuts or other damage, and replace it if necessary.

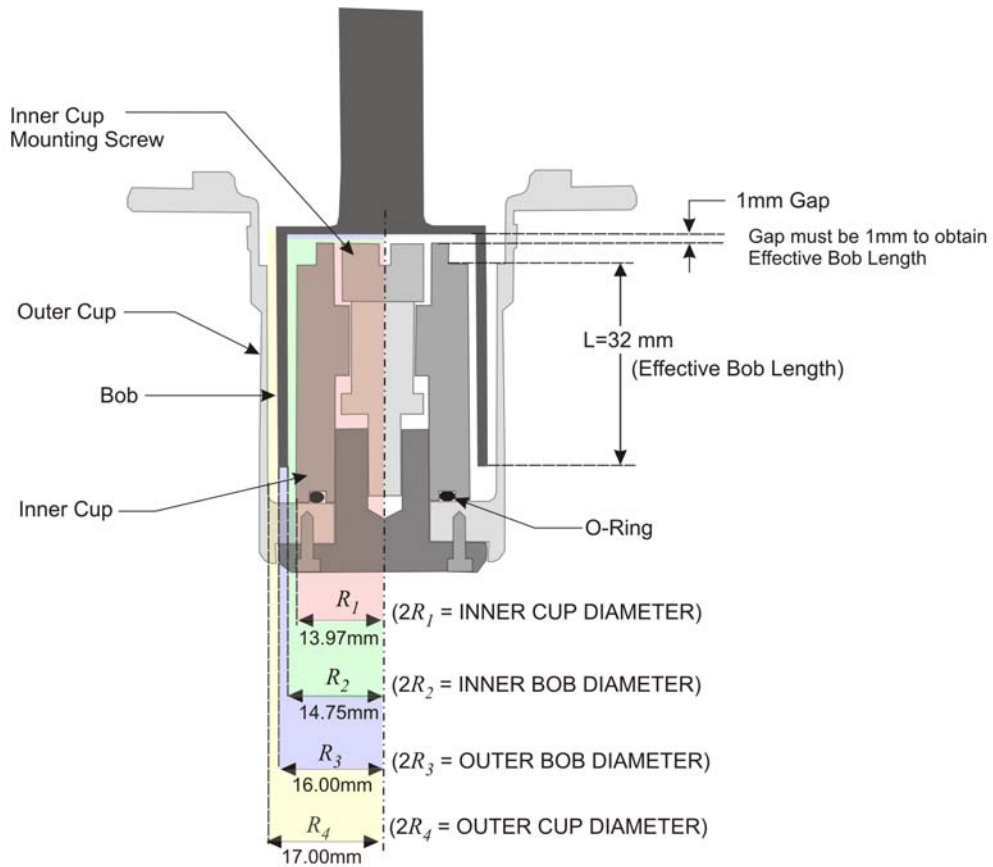


Figure 4-13. Fluid Bath 2 Double Wall Couette Setup Showing Inner and Outer Cup as Well as Tool Dimensions

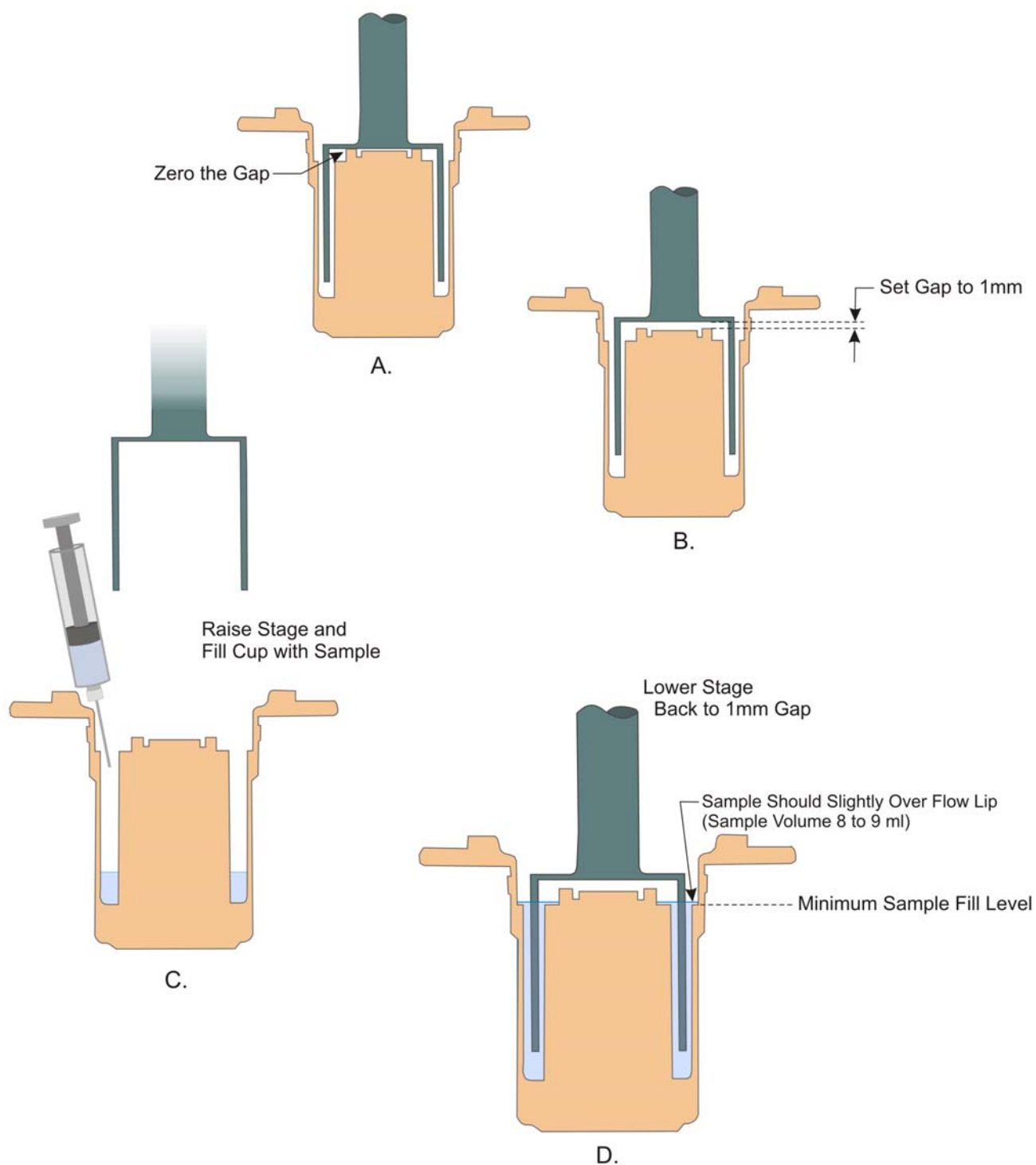


Figure 4-14. Fluid Bath 2 Double Wall Couette Sample Loading Procedures

A - Zero the gap; B - Set the gap to 1 mm;

C - Raise the stage and fill the cup; D - With sample loaded.

## Extensional Viscosity Fixture (EVF)

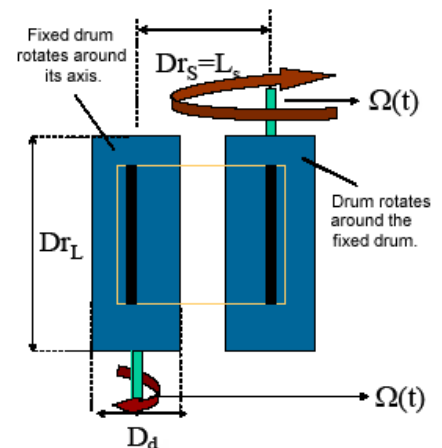
<p><b>Hencky Rate</b></p> $\frac{d}{dt} \epsilon_H = \frac{\Omega(t) \cdot D_d}{L_s}$	<p><b>Hencky Strain</b></p> $\epsilon_H(t) = \frac{d}{dt} \epsilon_H \cdot t$								
<p><b>Tensile Force</b></p> $F_s(t) = \frac{\text{Torque}(t)}{\frac{D_d}{2}}$	<p><b>Sample Area</b></p> $A_s(t) = A_0 \cdot \left( \frac{\rho_{\text{solid}}}{\rho_{\text{melt}}} \right)^{\frac{2}{3}} \cdot e^{-\epsilon_H(t)}$								
<p><b>Extensional Stress</b></p> $\sigma_E(t) = \frac{F_s(t)}{A_s(t)}$	<p><b>Extensional Viscosity</b></p> $\eta_E(t) = \frac{\sigma_E(t)}{\frac{d}{dt} \epsilon_H}$								
<p><b>Variables</b></p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%;">t = Time</td> <td style="width: 50%;">A<sub>0</sub> = Solid Area</td> </tr> <tr> <td>Ω(t) = Motor Speed</td> <td>σ<sub>solid</sub> = Solid Density</td> </tr> <tr> <td>D<sub>d</sub> = Drum Diameter</td> <td>σ<sub>melt</sub> = Melt Density</td> </tr> <tr> <td>L<sub>s</sub> = Sample Length</td> <td></td> </tr> </table>		t = Time	A <sub>0</sub> = Solid Area	Ω(t) = Motor Speed	σ <sub>solid</sub> = Solid Density	D <sub>d</sub> = Drum Diameter	σ <sub>melt</sub> = Melt Density	L <sub>s</sub> = Sample Length	
t = Time	A <sub>0</sub> = Solid Area								
Ω(t) = Motor Speed	σ <sub>solid</sub> = Solid Density								
D <sub>d</sub> = Drum Diameter	σ <sub>melt</sub> = Melt Density								
L <sub>s</sub> = Sample Length									
<p><b>Characteristics</b></p> <ul style="list-style-type: none"> <li>Cylindrical drums hold sample vertically between two clips.</li> <li>Can measure accurate elongation viscosity up to 10 s<sup>-1</sup></li> </ul>									
<p><b>Environmental Systems</b></p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%;">Ambient</td> <td style="width: 50%;">Oven</td> </tr> </table>		Ambient	Oven						
Ambient	Oven								



NOTE: Equations represent nominal values. Additional corrections are applied to account for variations of sample thickness with elongation.

### General Information

The Extensional Viscosity Fixture or EVF is an option for testing elongation viscosity of polymer melts and high viscosity viscoelastic fluids. The EVF design is based on the original Meissner concept to elongate the sample within a confined space by expelling the sample with rotary clamps. Instead of the rotary clamps, two cylinders are used to wind up the sample; one cylinder is rotating, the other measuring the force. See the figure to the right. In order to wind up the

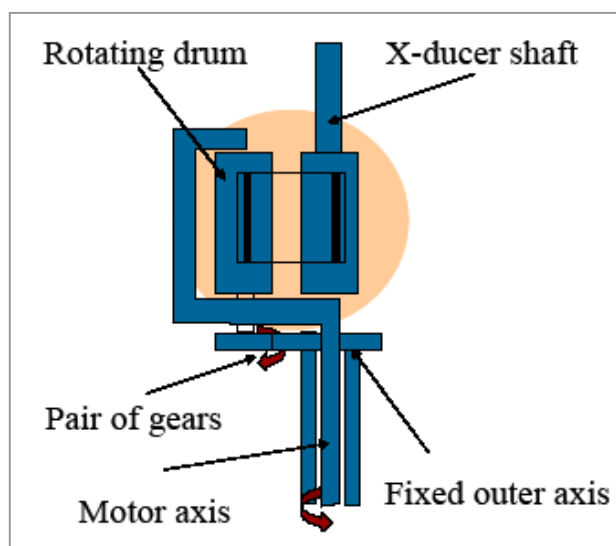


sample equally on both sides, the rotating cylinder moves on a circular orbit around the force-measuring cylinder while rotating around its own axis at the same time. Since the force measuring cylinder is fixed in space it can be directly coupled to the torque transducer of the ARES. The rotating cylinder movement is generated by the ARES actuator. As such the force measurement is decoupled from all the moving parts and consequently friction and inertia contributions are not affecting the material response, namely the force signal.

## Description of the Tool

The schematics of the Extensional Viscosity Fixture (EVF) are given in the figure to the right. Motor and transducer axis are aligned, with the rotating drum mounted eccentric at a distance (center to center) of 12.7 mm. In order to induce the spin in the rotating drum, a fixed hollow shaft, ending as a spur gear at the top, is mounted around the motor shaft to the ARES frame. As the eccentric mounted drum is orbiting around the transducer drum, the spur gear drives the rotation around its own axis. The EVF is designed to fit into the standard ARES oven. The diameter of the drums is 10.3 mm and the clearance between the drums is 2.4 mm.

The figure below shows the EVF option installed on the ARES rheometer. Since the cylindrical drums are mounted vertically, the sample is also loaded vertically onto the drums and attached with two tiny clips.



### Sample Support

On the EVF, the samples are mounted vertically and the sample length  $L_s$  is reduced to 12.7 mm (EVF). This means, that materials with a shear viscosity above 1000 Pa s are not significantly sagging under the effect of the gravity. Due to the small sample size and the fast heating rate of the ARES oven, the waiting time after loading the sample could be reduced to less than three minutes, thus preventing creep in the sample. During the measurement itself, the gravitational force does not contribute significantly.

## Preparing Samples

Sample preparation is an important criterion to obtain good data up to the maximum achievable Hencky strain. Sample non-uniformity and necking decrease the measured force, which shows as a viscosity decrease at large deformations. Sample homogeneity is a fundamental requirement for good elongation measurements up to Hencky rates of 5 and more. The goal of sample preparation is to make a homogeneous and stress free sample of the correct size.

The optimum sample size is recommended as follows:

- Length = 18 mm
- Width = 10 mm
- Thickness = 0.7 mm

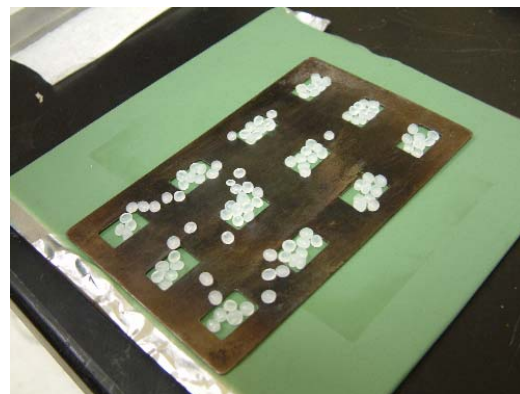
The best method to prepare samples is Hot Compression Molding (see the next section). The samples should be molded at a temperature above the test temperature and cooled slowly under pressure to obtain relaxed and stress free homogeneous samples with minimum dimensional variation. A compression mold (a 20-specimen mold for samples 18-mm long, 10-mm wide, and 0.7-mm thick) for polymers is part of the option.

**NOTE:** Samples with a high crystallinity content are difficult to mold stress free. Special care must be taken to anneal these samples before loading them into the instrument.

## Hot Compression Molding for EVF Samples

Molding conditions may vary depending on the melt temperature and relaxation properties of the materials being prepared. A basic method to use when molding samples for use with the EVF is described below:

1. Preheat the press to approximately 10 to 30°C above the temperature that will be used with your experiment.
2. Obtain a stiff upper and lower plate.
3. Cover the plate, if desired, with Teflon®-coated aluminum foil.
4. Place the molding plate on top of the foil.
5. Calculate the amount of sample needed then add 30 percent extra. (For the 20-specimen mold, assuming the density is 1 gram/cm<sup>3</sup>, you would need approximately 2.5 grams, plus 30 percent for a total of 3.3 grams.)
6. Spread the sample evenly into each hole as shown in the figure to the right.
7. Place the top plate over the sample and place the entire assembly into the press.
8. After the mold reaches the desired molding temperature, wait three to five minutes to allow the sample to be melted, then press the sample at about 10,000 pounds of pressure for a period of 10 to 15 minutes.
9. Turn off the heat and allow the sample to cool down with the pressure still applied.
10. Provide a sufficient pressing time to allow for stress relaxation. The sandwich of molding sheets, plates, and melt is either transferred to a cold press for slow cooling or simply solidified in the sample press by turning off the heating power.
11. Remove the samples when the temperature has cooled below the glass transition or melting point by pressing the samples out of the mold.



**NOTE:** In some cases (for example, branched or ultra-high molecular weight materials with a very long relaxation time), the melt has to be sheared for homogenization and elimination of the previous structure of the pellets prior to compression molding.

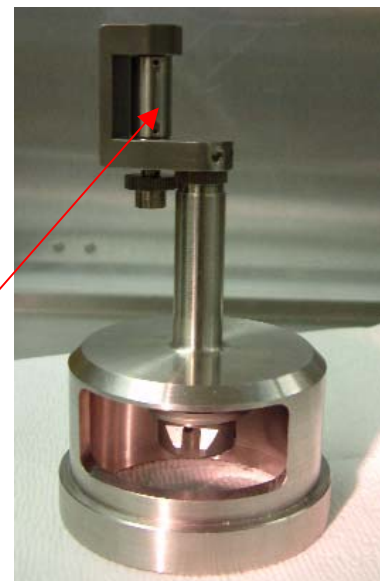
## Installing the Fixture

Follow the instructions below to install the Extensional Viscosity Fixture:

1. Fully open the oven and slide it as far to the left as it can go.
2. Push the manual stage control button on the side of the ARES to raise the stage.
3. Remove any current fixtures.
4. Clean the area around the lower fixture mount. Make sure you clean the outer and the bottom surfaces for the lower fixture to remove any dirt or debris. The figure to the right shows the motor without any fixtures installed.
5. Position the motor so that the knob is facing to the right as shown in the figure.
6. Select **Control/Motor Mode: Steady** to set the actuator to steady mode.
7. Turn off the motor power.
8. Position the lower fixture (see the figure to the right) with the drum on the left side of center and the clip facing toward the front. (There will be a locating pin to help you correctly position the fixture.)
9. Align the fixture, slide the safety cover up and mount the fixture. Place the mount ring over the fixture. Tighten the mount ring.
10. Mount the upper with sample clip facing front, but with a 30 degree offset to the right and tighten the knob. See the figure below.

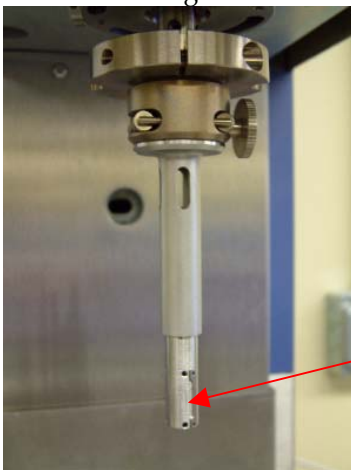


ARES Motor



Sample Clip

EVF Lower Fixture  
(Note: Safety Cover not shown)



Sample Clip

EVF Upper Fixture

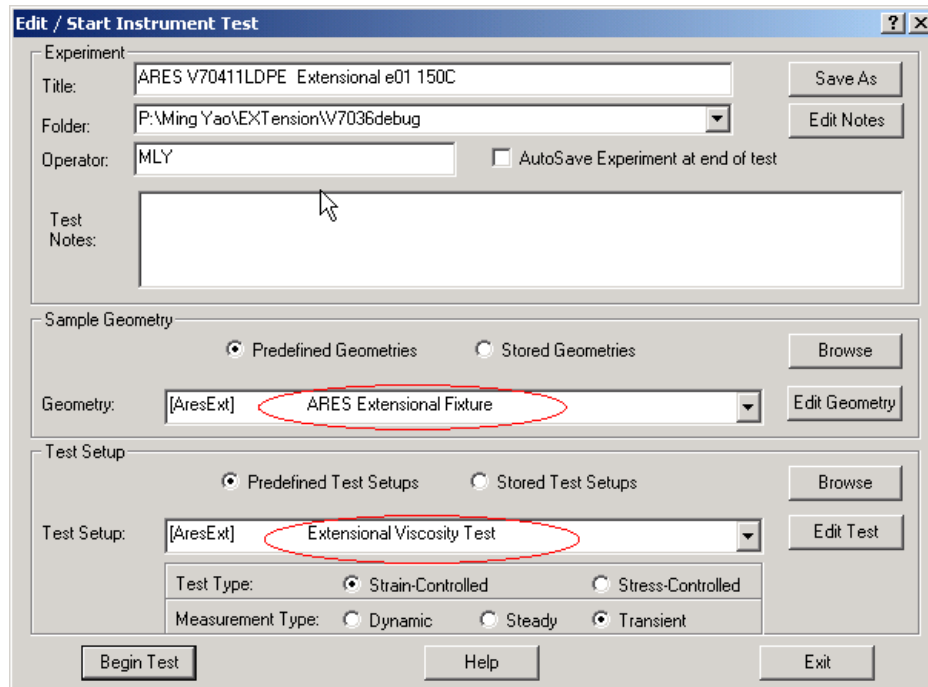
11. Use the manual button to lower the stage so that the upper and lower fixtures are close, but not touching. The distance between them should be less than 3 mm.
12. Select **Set Gap/Instrument Control** from the menu using the Orchestrator software. The **Gap/Instrument Control Panel** is displayed.
13. Zero the Torque and Normal by pressing the **Offset Torque To Zero** and the **Normal To Zero** buttons.
14. Enter 0.5000 as the commanded gap.
15. Set the gap by pressing the **Set Zero Gap** button.
16. Confirm the position of the two sample clips then pull the clips out a little to accommodate sample loading after the instrument has been fully prepared.
17. Turn on the motor. (See page 36 for information if needed.)



## Preparing the Instrument and Test

After the upper and lower fixtures have been correctly installed, follow these steps to prepare the instrument for the sample:

1. Close the ARES oven completely and latch it.
2. Select **Utilities/Service/Instrument Configuration** to display the **Setup Instrument Options** dialog.
3. Select “Mode 3. RAA Oven Air Temp” as the **Temperature Loop Control**.
4. Access the **Instrument Control Panel** dialog. Select **On** for the **Environmental Controller** option and enter the desired **Temperature** to be used for your experiment.
5. Click the green **Start** button. The oven will begin preheating and the **Edit/Start Instrument Test** dialog is displayed. Perform the following steps to set up your test using this dialog shown below.



- a. Enter the desired **Title**, **Folder**, **Operator**, and **Test Notes**. Check **AutoSave Experiment at end of test**, if desired.
- b. Select **Predefined Geometries** and choose “ARES Extensional Fixture” from the **Geometry** drop-down list.
- c. Measure the sample dimensions (width and thickness). Click the **Edit Geometry** button. Enter the dimensions in the appropriate field. Click **OK**.
- d. Click the radio button, **Predefined Test Setups** and select the “Extensional Viscosity Test.”

**NOTE:** The EVF tool can only be used with the Extensional Viscosity Test.

- e. Click the **Edit Test** button to display the **Extensional Viscosity Test** dialog shown in the figure on the next page.

**Extensional Viscosity Test**

Temperature ..... 150.0 [°C] Max=600.0°C Min=20.0°C

Sampling Mode .  Log  Linear

Points Per Zone 200 [ ] Max=350 Min=20

---

Extension Rate ... 0.1 [1/s] Max=81.10236 Min=8.11e-04

Extension Zone 1 2

Time [s or h:m:s] 35 0

---

Solid Density ..... 0.919 [g/cm<sup>3</sup>]

Melt Density ..... 0.781 [g/cm<sup>3</sup>]

Prestretch Rate ..... 0.005 [1/s] Max=81.10236 Min=8.11e-04

Prestretch ..... 0.71 [mm] / 11.1 [s]

Relaxation after Prestretch 40.0 [s or h:m:s]

Options: Delay:Off

Ok Options End of Test Save As Help Cancel

- f. Enter the desired **Temperature** for your test.
- g. Click on the **Log** radio button as the **Sample Mode** and enter the desired **Points Per Zone** (e.g., 200).
- h. Enter the **Extensional Rate**.
- i. Enter the **Extension Zone Time** in the Zone 1 field. This value is normally 3.5 to 4.0/Extensional Rate. In this example 35 has been used.
- j. Enter the literature sample **Solid Density** at room temperature and the **Melt Density** at testing temperature.

**NOTE:** Densities are used to calculate the actual dimensions of the sample at test temperature.

- k. Enter the **Prestretch Rate**. This value is normally 0.005 to 0.01 sec<sup>-1</sup>. The **Prestretch** will be calculated automatically.

**NOTE:** Prestretch is used to compensate for the thermal expansion of the sample when it is heated up from room temperature. When the densities are entered, the software will calculate how long the sample should expand and determine the Prestretch length and time automatically.

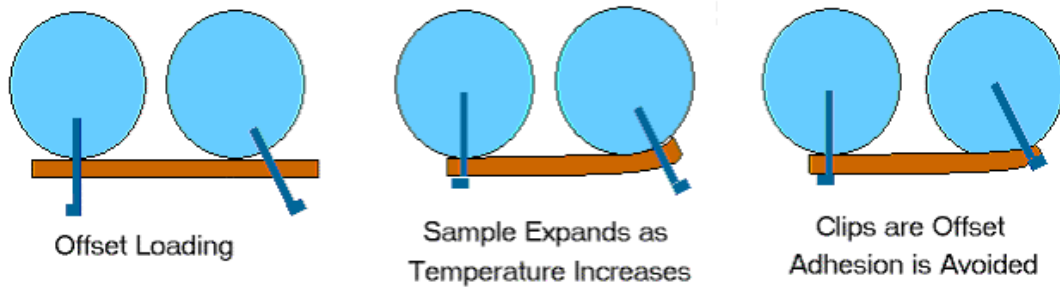
- l. Enter the **Relaxation after Prestretch** time to allow the sample to enter relaxation after it has been prestretched.
- m. Click the **Options** button. Enter the desired **Delay Time**, which depends upon the sample being tested. Check the function **Automatically Start on Temp.** to begin the test as soon as the desired temperature has been achieved. Click **OK**.
- n. Click **OK** again to exit the **Extensional Viscosity Test** dialog and return to the **Edit/Start Instrument Test** dialog.
- o. Proceed to the next section to load your sample.



## Loading Samples

The quality of your test results when using the EVF depends significantly on the uniformity of the sample and the careful loading of the sample onto the fixture. During a test the sample is going to expand as the temperature rises, which can result in the sample adhering to the fixture drum, reducing its length and increasing the force.

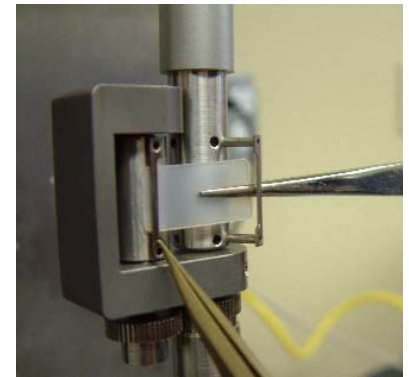
Follow the instructions below to use an offset technique when loading the sample (as seen in the figure below) in order to avoid affecting your test results adversely.



Correct Offset Loading Technique

### Loading the Sample Using the Offset Technique

1. Check the oven temperature. When it has reached the desired testing temperature, open the oven carefully.
2. Thread the sample (prepared as directed previous in this section) from the right side to the left side through the two opened sample clips. See the figure to the right.
3. Close the left clip just enough to touch the sample. Do not press it in too tightly and compress the sample end.
4. Close the right clip using the same technique. Do not compress the sample.
5. Close the oven.



Loading the EVF Sample

## Running Experiments

Once you have set up the test and correctly prepared and loaded the sample, follow these steps to run your experiment.

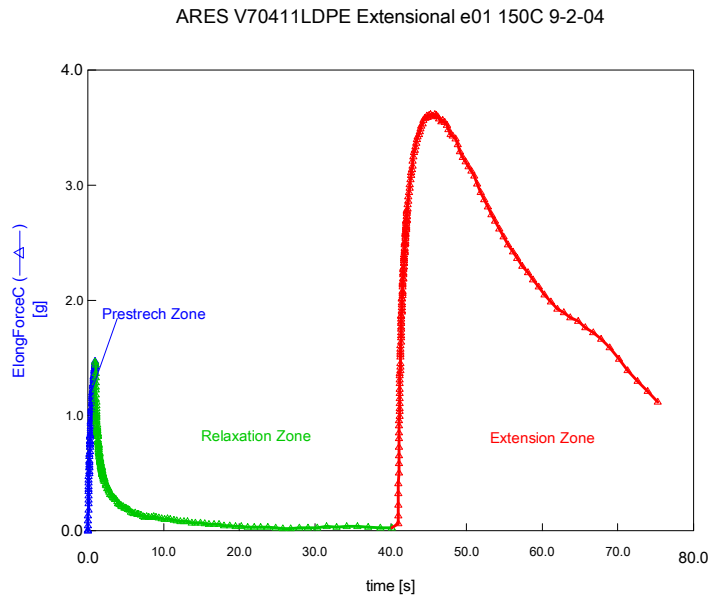
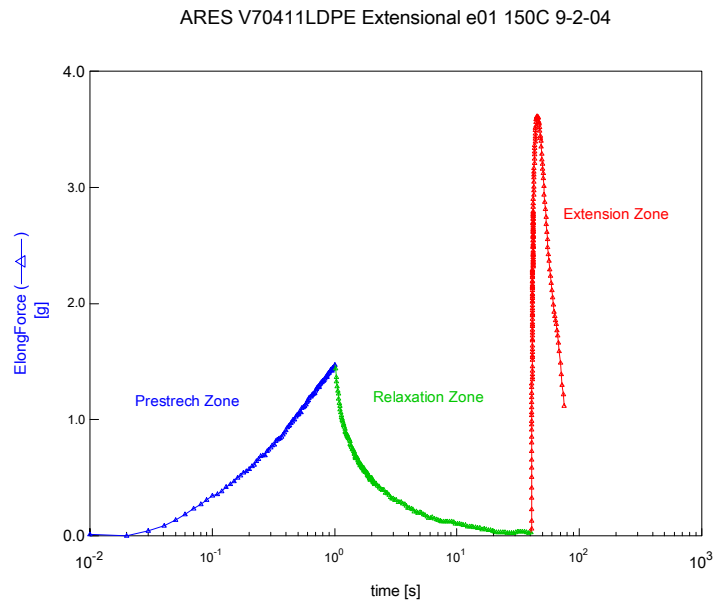
1. Monitor the temperature. When the oven has reheated up to the testing temperature, click the **Begin Test** button on the **Edit/Start Instrument Test** dialog. The experiment will start using the parameters you set up previously.
2. Save the data when the test has been completed, if you have not selected **AutoSave** as part of your setup procedure.
3. Allow the oven to cool the sample to ambient temperature.
4. Turn off the motor.
5. Open the oven.

6. Unload the sample. Make sure the sample has solidified once more, then peel the sample from the drum and sample clips. In some cases you may need to remove the sample at a slightly elevated temperature or use a scraping tool made of soft metal such as brass or aluminum.
7. Position the fixture to prepare for the next experiment.

## Plotting Data

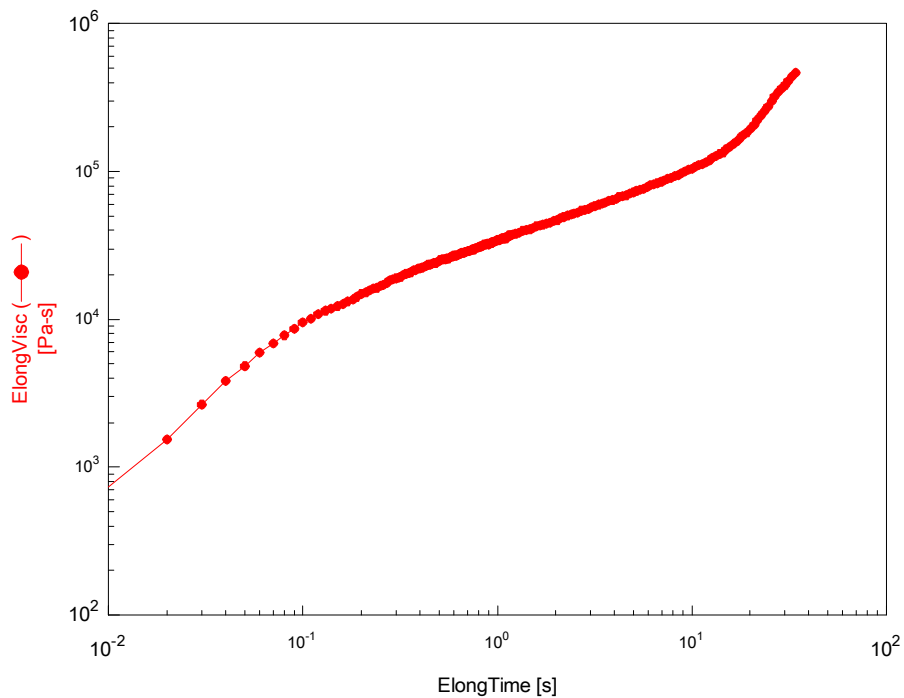
Follow the recommendations below to plot EVF data.

1. Plot ElongForec versus Time to monitor the whole experiment. As shown below, there are three zones: Prestrech, Relaxation and Extension in log scale or linear scale of Time.

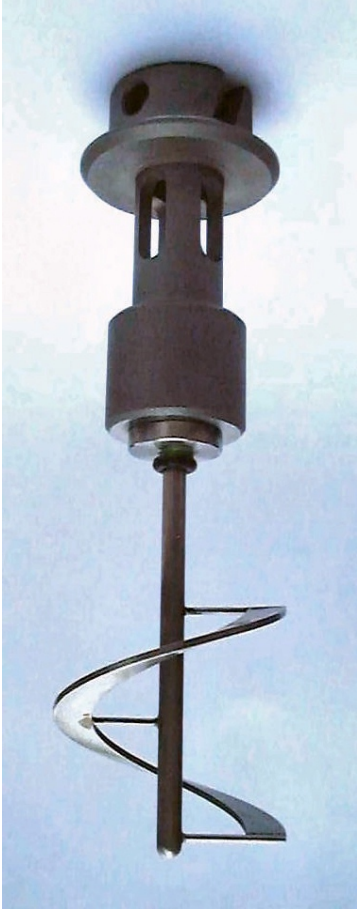


2. Plot ElongVisc versus ElongTime after the measurement as shown below, which gives the final result of extensional viscosity data.

ARES V70411LDPE Extensional e01 150C 9-2-04



## Helical Ribbon

<p><b>Strain Constant</b></p> $K_\gamma = \dot{\gamma} / 2\pi N$ $K_\gamma = 2.46 \text{ 1/rad}$	<p><b>Stress Constant</b></p> $K_\tau = \tau / T$ $K_\tau = 2.68 \text{ E4 Pa/Nm}$	<p style="text-align: center;">Helical Ribbon Fixture</p> 
<p><b>Variables</b></p> <p><math>K_\tau</math> = Stress Constant  <math>K_\gamma</math> = Strain Constant  N = rotational speed  T = torque</p>		
<p><b>Characteristics</b></p> <ul style="list-style-type: none"> <li>• Designed for use with the 34 mm cup</li> <li>• Stress and strain constants calculated using Couette analogy</li> <li>• Prevents sedimentation of samples</li> <li>• Can be used for measurements of materials with large particles</li> <li>• Can be used to mix multiphase systems (solids or liquids)</li> </ul>		
<p><b>Options</b></p> <p>A narrow high speed mixer or additional sensors (user supplied) can be used in conjunction with the helical ribbon, since the ribbon is attached to the stationary transducer while the cup is rotating or oscillating. Note, that the calibration constants change slightly with additional elements are immersed in the fluid. Recalibration might be necessary.</p>		
<p><b>Environmental Systems</b></p> <p>Fluid Bath, Fluid Bath 2 *</p> <p>* See Chapter 2 for more details regarding the Bath.</p>		

## General Information

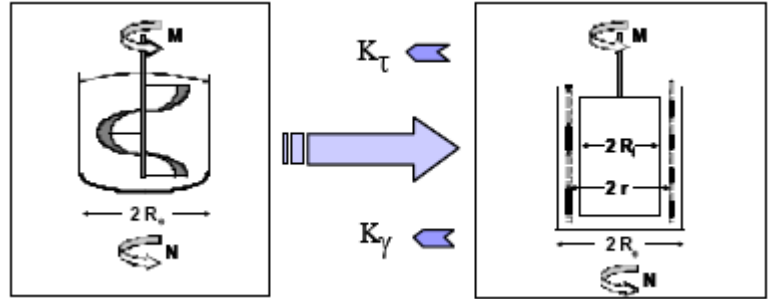
The helical ribbon fixture is a mixing element, used with the standard 34 mm cup to test complex fluids. With the helical ribbon fixture, the rheometer can be operated as an instrumented batch reactor to measure the rheological properties under real flow conditions (systemic rheology => the rheology of the system including material and environment), while even conducting complementary measurements such as conductivity measurements.

Since the flow is complex and the material usually behaves in a non-linear fashion, the calculation of a true or representative deformation, or deformation rate, for the mixing element is an issue. Based on a Couette

analogy, average deformation and stress coefficients can be determined, which relate the motor rotation speed to the material deformation rate and the measured torque to the stress.

### Couette Analogy

The couette analogy assumes the helical ribbon mixing device, in a mixing chamber, to be a virtual cylindrical bob in a cylindrical chamber as shown in the figure to the right. The analogy consists of determining an equivalent inner cylinder with a radius  $R_i$  and the same height as the impeller, which generates the same torque,  $M$ , at the same rotational speed,  $N$ . The outer cylinder of the rheo-reactor has the same radius as the outer cylinder of the equivalent concentric cylinder system.



Since the equivalent inner radius varies only slightly with the power law index,  $n$ , a calibration procedure can be used for the mixing element using a Newtonian or a well-characterized power law fluid.

Once  $R_i$  has been obtained, shear rate and shear stress can be calculated as a function of the radius  $r$ .

Ait-Kadi, *et al.* have shown, that at a given radius  $r^*=(R_i+R_e)/x^*$ , where  $x^*=2$  for small gaps ( $R_i/R_e>0.9$ ) the shear rate is essentially independent of the nature of the fluid, *i.e.*, independent of the local power law index. For large gaps  $x^*$  becomes greater than 2.

### Calibration

The calibration consists of determining the stress and strain geometry constants for a given reactor set up. After calibration, it is important to use the same amount of fluid and immerse the helical ribbon to the same position as during the calibration.

Following are the calibration steps:

1. Measure  $\tau$ ,  $\eta$ ,  $d\gamma/dt$  for a Newtonian fluid (1000cP silicone oil) using standard 34-32 mm Couette.
2. Replace the inner cylinder with the new mixing element and measure the torque  $T$  at the same angular rotation speed for the same fluid as in step 1.
3. Calculate  $R_i$  assuming a power law fluid:

$$\eta = k\dot{\gamma}^{n-1} \quad (n=1 \text{ for the Newtonian silicone oil}):$$

$$R_i = \frac{R_e}{\left[ 1 + \frac{4\pi N}{n} \left( \frac{2\pi k L R_e^2}{T} \right)^{1/n} \right]^{n/2}}$$

4. Calculate the stress  $\tau(r)$  and  $d\gamma(r)/dt$ .

$$\tau = \frac{T}{2\pi L r^2} \quad \text{and} \quad \dot{\gamma} = \left[ \frac{\frac{4\pi}{n} \left(\frac{R_i}{r}\right)^{2/n}}{1 - \left(\frac{R_e}{R_e}\right)^{2/n}} \right] N$$

For a Newtonian fluid use  $n=1$  and for small gaps  $r = (R_i + R_e)/2$

5. Calculate the stress and strain constants:

$$K_\tau = \tau/T \quad \text{and} \quad K_\gamma = \gamma/2\pi N$$

NOTE: For a large gap,  $r$  is determined experimentally.

Reference : A. Aït-Kadi, P. Marchal, A.S. Chrissemant, M. Bousmina and L. Choplin, *Can.J.Chem.Eng.*, **80:6**, 1166 (2002)

## Operating Ranges

The operation range for stress and strain is the minimum and maximum of the instrument torque and displacement specification multiplied with the stress or stress constant. The modulus and viscosity are calculated as given in the Couette section (see page 181), however with fixed constant values.

## Tool Installation

For installation of the lower tool into either of the ARES fluid bath temperature systems, please refer to the appropriate section of Chapter 2 (for your specific bath) for instructions.

1. Select the **Set Gap/Instrument Control** function under the Control menu in Orchestrator. Use the "Send to Top" button to raise the stage to the loading position.
2. Verify that the motor is on.
3. Mount the upper tool on the transducer shaft and lower the tool completely into the fluid bath.
4. Using the **Set Gap/Instrument Control** function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset Torque to Zero" buttons.

## Sample Loading

In general, pour the sample into the cup then lower the helical ribbon to the vertical position (gap) used during calibration. If the entire ribbon is not below the surface of the sample, adjust the sample volume accordingly. Nominal sample volume is as follows:

Cup size	Sample Volume
34 mm	15 ml

# Test Formulas

All formulas and geometry constants are also listed in Orchestrator Online Help (see *Reference Guide* under the *Contents* tab).

## Dynamic Measurement Formulas

Table 4-1. Dynamic Measurement Formulas

VARIABLE AND FORMULA	DEFINITION OF VARIABLES
$\tau = \text{STRESS}$ $= (K_{\tau}) (M)$	$K_{\tau} = \text{Stress Constant}$ $M = \text{Torque (g}\cdot\text{cm)}$
$\gamma = \text{STRAIN}$ $= (K_{\gamma}) (\theta)$	$K_{\gamma} = \text{Strain Constant}$ $\theta = \text{Shearing angle of motor (radians)}$
$G' = \text{ELASTIC (STORAGE) MODULUS}$ $= \cos \delta \left( \frac{\tau}{\gamma} \right)$	$\delta = \text{Phase angle (phase shift between stress and strain vectors)}$
$G'' = \text{VISCOUS (LOSS) MODULUS}$ $= \sin \delta \left( \frac{\tau}{\gamma} \right)$	
$G^* = \text{COMPLEX MODULUS}$ $= \sqrt{(G')^2 + (G'')^2} = \frac{\tau}{\gamma}$	
$\tan \delta = \text{LOSS TANGENT}$ $= \frac{G''}{G'}$	
$\eta' = \text{REAL PART OF DYNAMIC (COMPLEX) VISCOSITY}$ $= \frac{G''}{\omega}$	$\omega = \text{Frequency (angular, in rad/sec)}$
$\eta'' = \text{IMAGINARY PART OF DYNAMIC (COMPLEX) VISCOSITY}$ $= \frac{G'}{\omega}$	

Table 4-1. Dynamic Measurement Formulas (Continued)

VARIABLE AND FORMULA	DEFINITION OF VARIABLES
$\eta^* =$ DYNAMIC (COMPLEX) VISCOSITY $= \frac{G^*}{\omega}$	
$J' =$ STORAGE COMPLIANCE $= \frac{G'}{(G')^2 + (G'')^2}$	
$J'' =$ LOSS COMPLIANCE $= \frac{G''}{(G')^2 + (G'')^2}$	

## Steady and Transient Measurement Formulas

Table 4-2. Steady and Transient Measurement Formulas

PARAMETER AND FORMULA	DEFINITION OF VARIABLES
$\tau =$ STRESS $= (K_\tau) (M)$	$K_\tau =$ Stress Constant $M =$ Torque (g•cm)
$\gamma =$ STRAIN $= (K_\gamma) (\theta)$	$K_\gamma =$ Strain Constant $\theta =$ Shearing angle of motor (radians)
$\dot{\gamma} =$ STRAIN RATE (SHEAR RATE) $= (K_\gamma)(\dot{\theta})$	$K_\gamma =$ Strain Constant $\dot{\theta} =$ Angular velocity of motor (radians/sec)
$\eta =$ VISCOSITY $= \frac{\tau}{\dot{\gamma}}$	$\dot{\gamma} =$ STRAIN RATE $= \frac{d\gamma}{dt}$
$N_f =$ NORMAL STRESS $= K_Z * F_Z$	$K_Z =$ Normal stress constant $F_Z =$ Normal force (g)



Table 4-3. Strain and Stress Constants

Geometry	Strain Constant $K_\gamma$	Stress Constant $K_\tau$
Parallel Plates	$\frac{R}{H}$	$\frac{2(G_c)}{\pi\left(\frac{R}{10}\right)^3}$
Cone and Plate	$\frac{1}{\beta}$	$\frac{3000(G_c)}{2\pi R^3}$
Torsion Rectangular	$K_\gamma = \frac{T(1/3 - 0.21T/W)(3W + 1.8T)}{W*L}$	$1000 \left( \frac{3 + \left(1.8\left(\frac{T}{W}\right)\right)}{WT^2} \right) G_c$
Fiber	$\frac{1}{2}$	$\frac{100}{1.8756\pi R^2} G_c$
Film	$\frac{1}{2}$	$\frac{100}{1.8756TW} G_c$
Couette	$\frac{2}{1 - \left(\frac{R_B}{R_C}\right)^2}$	$\frac{(1000)(G_c)}{2\pi L(R_B)^2}$
Double Wall Couette	$\frac{1}{\left(\frac{R_2}{R_1}\right)^2 - 1} + \frac{1}{1 - \left(\frac{R_3}{R_4}\right)^2}$	$\frac{(1000)(G_c)}{2\pi L\left((R_2)^2 + (R_3)^2\right)}$
<p>Variables:  <math>L</math> = length (mm)    <math>W</math> = width (mm)    <math>T</math> = thickness (mm)    <math>R</math> = radius (mm)    <math>H</math> = height (mm)  <math>G_c</math> = gravitational constant = 980.7 cm/s<sup>2</sup>    <math>\beta</math> = cone angle  <math>R_B</math> = radius of the bob    <math>R_C</math> = radius of cup                      For double wall Couette refer to Figure 4-12 for definition of <math>R_1, R_2, R_3, R_4</math></p>		



---

---

# Chapter 5

## Calibration

### Introduction

This chapter contains calibration procedures that you can performed. Do not attempt any calibration procedure unless you are thoroughly familiar with the operation of both the instrument and Orchestrator software. Calibration procedures are given for Orchestrator version 6.5.6, which is the software released with the instrument at the time of this manual publication. However, menu and function names may change without notice during subsequent software releases.



**WARNING: HIGH VOLTAGE** is used in the operation of this instrument. **DEATH ON CONTACT** may result if operating personnel fail to observe safety precautions. Learn the areas of high voltage connections, and exercise care not to contact these areas when performing instrument calibration. Prior to working inside the instrument, remove all jewelry, turn off the power, and ground points of high voltage before touching them. Make adjustments using an insulated electronic adjustment tool. Do not make physical contact with any component inside the instrument while power is applied to the instrument.

---

# Procedures

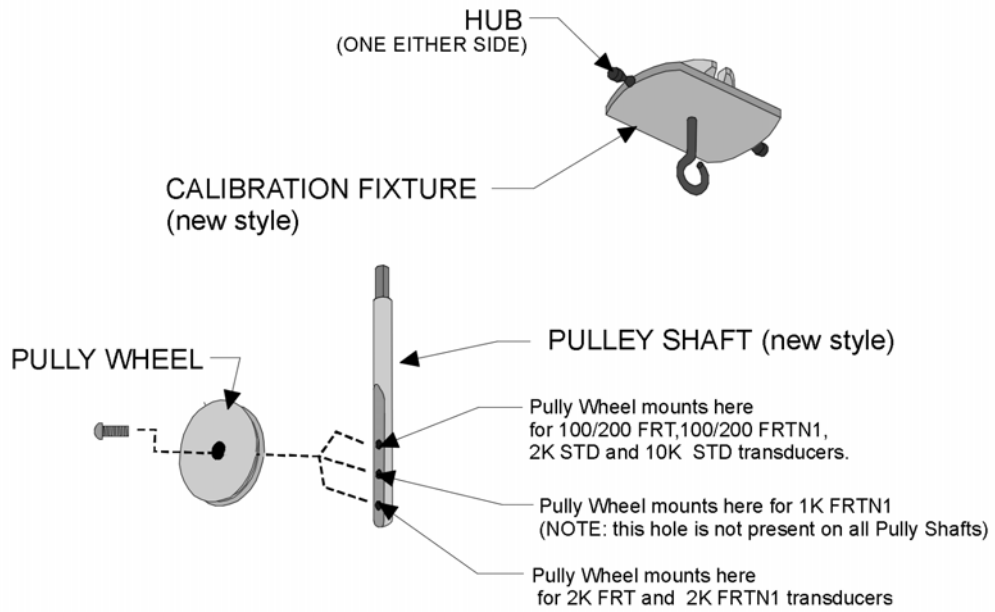
## Calibration Intervals

Table 5-1 lists required calibration tasks and the recommended calibration interval for each task. The table also shows when certain calibrations must be performed following repairs.

Table 5-1. Calibration Tasks and Recommended Intervals

CALIBRATION TASK	CALIBRATION INTERVAL
Torque Calibration	Suggested: Monthly. Mandatory: Following transducer replacement.
Normal Force Calibration	Suggested: Monthly. Mandatory: Following transducer replacement.
Phase Angle Check	Suggested: Monthly. Mandatory: Following transducer replacement. When attempting to diagnose system problem.
Strain Calibration Check	Suggested: Monthly Mandatory: When attempting to diagnose suspected motor control system problems .

## A. NEW STYLE CALIBRATION FIXTURE



## B. OLD STYLE CALIBRATION FIXTURE

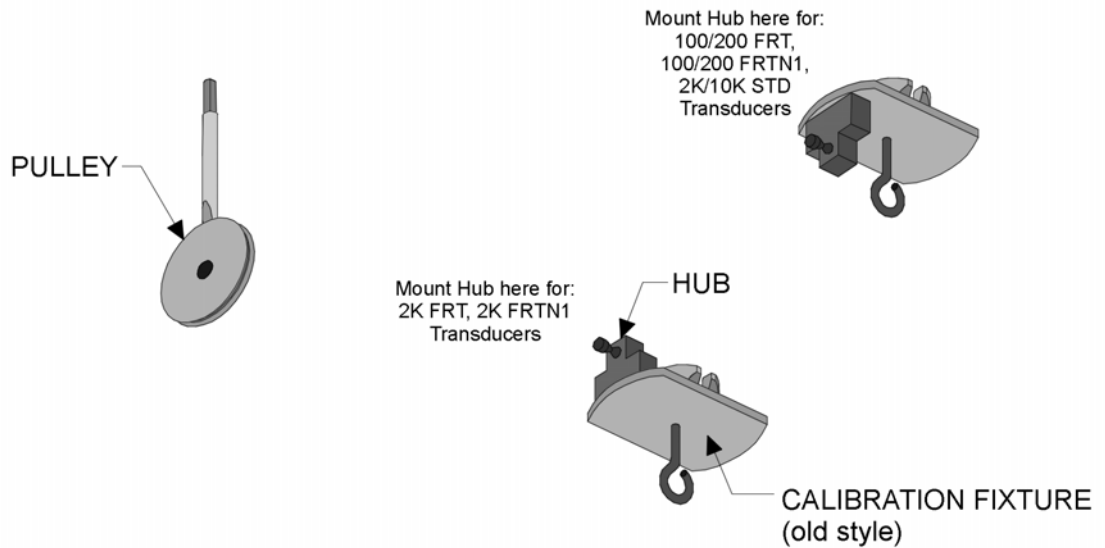


Figure 5-1. ARES Calibration Tools

## Torque Calibration (for All Transducers)

Torque Calibration ensures that the transducer is accurately measuring torque. The calibration involves hanging a precision weight on the calibration tool, a 2.5-centimeter moment arm that is mounted on the transducer during calibration. The applied torque is therefore the product of the weight and the 2.5-centimeter moment. For example, hanging a 500 gram weight applies a torque of  $(500 \text{ g})(2.5 \text{ cm}) = 1250 \text{ g}\cdot\text{cm}$ .

**NOTE:** For this, and all subsequent calibration procedures, the instructions assume that the Host Computer is connected to the ARES test station, and that Orchestrator is running.

### Procedure

1. If calibrating an FRT, ensure that the transducer is set to High Range before proceeding. To set the transducer to High Range do the following:
  - a. Access the *Set Transducer Characteristics* form (Figure 5-7) by selecting the *Transducer* option from the *Service* function of the Utilities pull down menu.
  - b. Using the "Transducer Selected" menu, select the high range transducer then Click "Ok".
2. Remove any test tools.
3. Turn on the motor.
4. Raise the stage to bring the bottom of the stainless steel transducer cover about 2 inches below the instrument cover (Figure 5-2).
5. Determine which calibration tool you have based upon Figure 5-1. Depending upon your tool, and transducer, mount the pulley wheel, or hub, appropriately.
6. Install the calibration tool and pulley as shown in Figure 5-2.
  - a. For 10K STD Transducers: Two pulleys are supplied. Referring to Figure 5-2, insert a pulley into each side of the Test Station frame, ensuring that the flat machined into each pulley shaft faces the access hole. Please note that Figure 5-2 shows the pulley on the right side only.
  - b. For all other transducers: Insert the pulley into the Test Station frame as shown in Figure 5-2, ensuring that the flat machined into the pulley shaft faces the access hole.Secure the pulley(s) by tightening the setscrew in the access hole (Figure 5-2) using a 1.5-mm hex wrench. Ensure that the calibration tool and pulley are installed as shown in Figure 5-3. If you have a 10K STD transducer, an additional pulley should be installed on the left.
7. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force and torque on the motor using the "Offset Normal Force to Zero" and "Offset Torque to Zero" buttons. Exit the form when finished.
8. Prepare the calibration line specified in Table 5-2, depending on the transducer in use. Prepare the calibration line(s) by making a loop at each end.

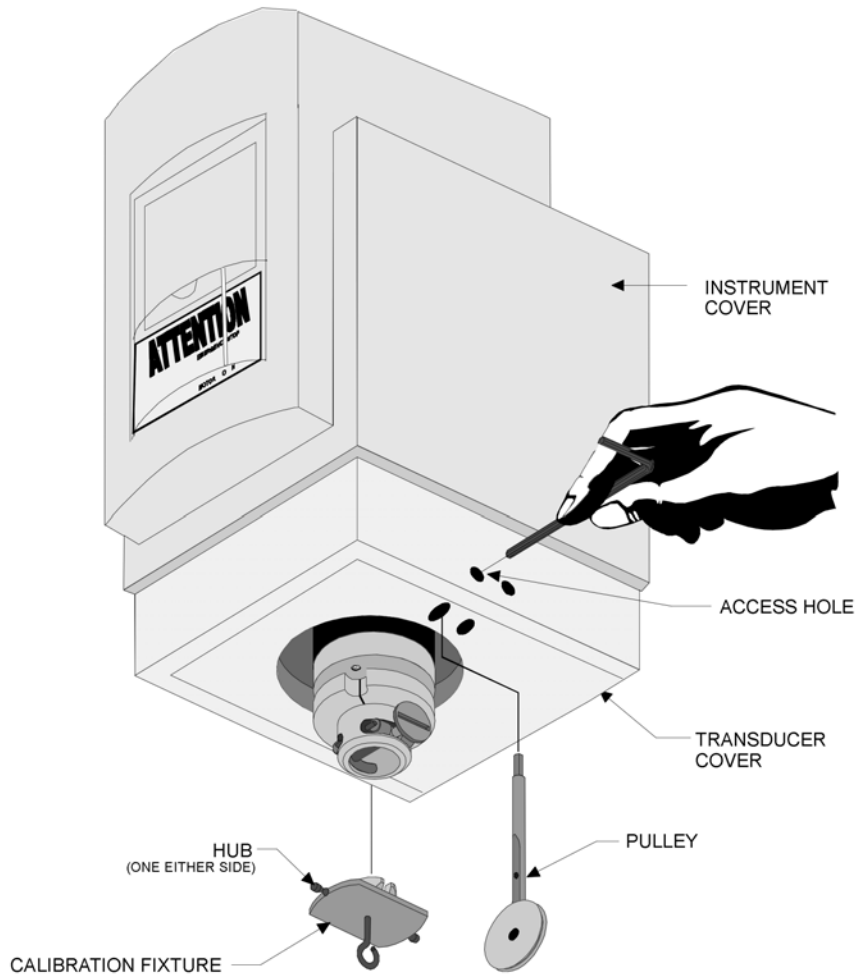


Figure 5-2. Installation of the Calibration Tool and Single Pulley

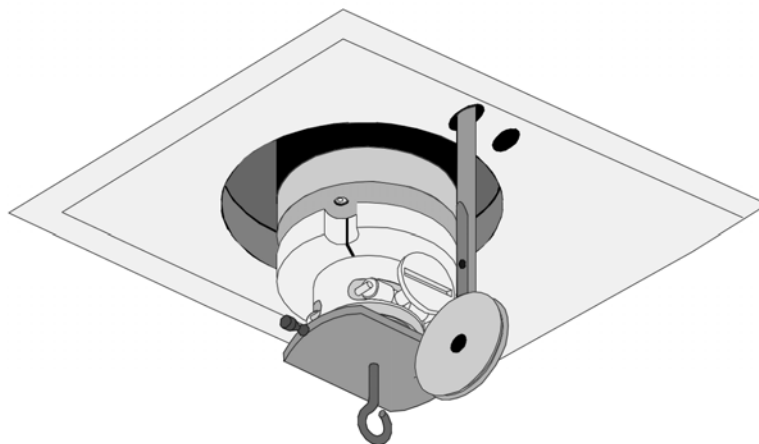


Figure 5-3. Calibration Tool and Single Pulley Installed

Table 5-2. Calibration Lines

TRANSDUCER	CALIBRATION LINE
2K FRTN1 2K FRTN1E 1K FRTN1 2K STD	Cut one length monofilament line (part number 613-01075)
100 FRTN1 200 FRTN1 100 FRT 200 FRT	Cut one length of thread (part number 613-00716)
10K STD	Cut two separate lengths of monofilament line (part number 613-01075)

9. Access the *Transducer Characteristics* form using the Orchestrator function *Calibrate Instrument* under the Utilities pull down menu.
10. Select the "XducerCal" button. Establish a zero torque reference value by selecting the "Zero" button. Wait about 30 seconds, during which time the instrument takes several readings to establish a zero normal reference. When zeroing is completed, the *Transducer Calibration* form is displayed (Figure 5-4). The zero value displayed in this form should be less than 0.1% of the full scale Torque value.

If, after selecting the "ZERO" button, the TORQUE value displayed is either very high (such as 1E+5) or exactly zero, refer to the Troubleshooting Guide.

**NOTE:** Do not hang any weights until after at least one (1) zero reading has been taken.

11. Apply a calibrated torque (Figure 5-5), depending on your transducer, as follows:
  - a. Place one end of the line over the hub on the calibration tool, and place the line in the groove of the pulley.
  - b. Hang the weight specified in Table 5-3 (depending on the transducer in use) from the loop in the line.
  - c. Ensure that the weight(s) is (are) free to hang without obstruction, and that the weight is steady (not swinging from side-to-side).
12. Select the "Torque Cal" button. The *Torque Calibration* form is displayed.
13. Enter the Calibration Torque specified in Table 5-3, depending on the transducer in use.
14. Select the "Calibrate Now" button. When calibration is complete, the *Transducer Calibration* form is again displayed.



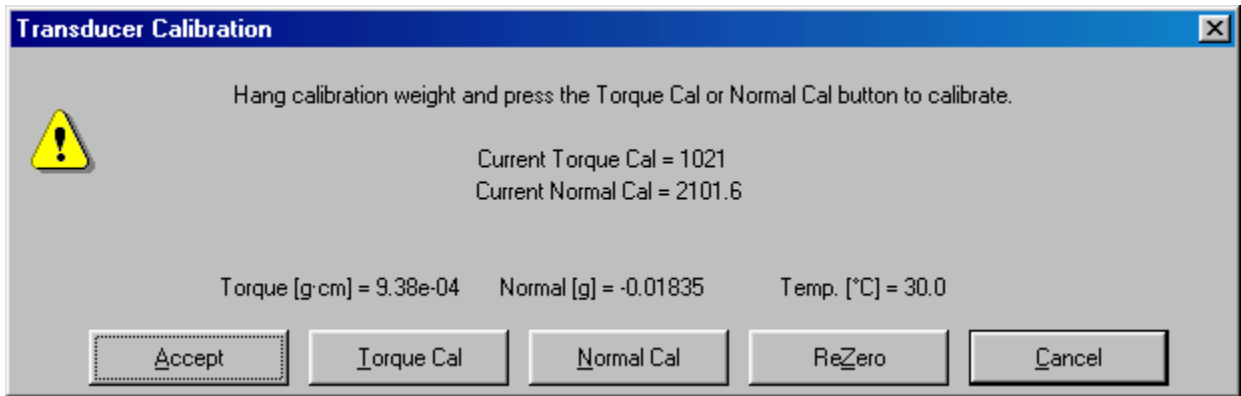


Figure 5-4. Transducer Calibration Form

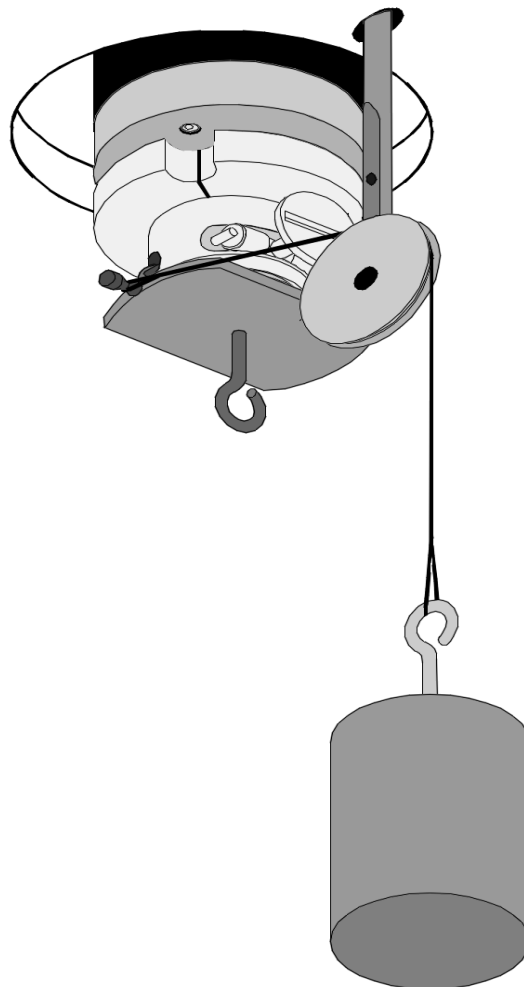


Figure 5-5. Applying Torque Using the Calibration Weight

15. Verify that the **Torque** value displayed on the *Transducer Calibration* Form (Figure 5-4) is within the limits for the TORQUE VALUE shown in Table 5-3, depending on the transducer in use. Verify that the **Current Torque Cal** value displayed in the *Transducer Calibration* Form is within the limits for the CALIBRATED FULL SCALE VALUE shown in Table 5-3.

If the Torque values are not within the specified limits, contact TA Instruments Service.

16. Select the "Accept" button. Control returns to the *Transducer Characteristics* form.
17. Verify that the **Torque Calibration Value** displayed in the form (the high range value if the transducer is an FRT) is the same as the **Current Torque Cal** value just displayed in the *Transducer Calibration* form. Press "Ok".
18. If the transducer in use is an FRT perform the following step. If the transducer is a STD transducer, go to step 19.
  - a. Access the *Set Transducer Characteristics* form (Figure 5-7) by selecting the *Transducer* option from the *Service* function of the Utilities pull down menu.
  - b. Divide the displayed *Torque Calibration Value* for the high range transducer by 10 then enter this value (into the form) as the *Torque Calibration Value* for the low range transducer. For the 1K FRTN1 transducer only, divide the high range *Torque Calibration Value* by 50 and enter this value for the low range transducer setting.
19. Click Ok. Proceed to the Normal Force Calibration if desired. If Normal Force Calibration is not to be performed, remove the calibration tool, weight, and pulley, and store them in the calibration kit.

This concludes the Torque Calibration.

Table 5-3. Torque Calibration Weights, Applied Torques, and Full Scale Values

TRANSDUCER	WEIGHT	CALIBRATION TORQUE (applied)	TORQUE VALUE (displayed)	CALIBRATED FULL SCALE VALUE (computed)
2K FRTN1 2K FRTN1E 2K STD	200 grams (p/n 613-01221)	500 g•cm	500 ± 2 g•cm (498 to 502)	2100 ± 5% (1995 to 2205)
1K FRTN1	200 grams (p/n 613-01221)	500 g•cm	500 ± 1 g•cm (499 to 501)	1050 ± 5% (997.5 to 1102.5)
100 FRTN1 100 FRT	20 grams (p/n 613-02775)	50 g•cm	50 ± 0.1 g•cm (49.9 to 50.1)	105 ± 5% (99.75 to 110.25)
200 FRTN1 200 FRT	20 grams (p/n 613-02775)	50 g•cm	50 ± 0.2 g•cm (49.8 to 50.2)	210 ± 5% (199.5 to 220.5)
10K STD	Two - 1000 gram (p/n 613-01222)	5000 g•cm	5000 ± 10 g•cm (4980 to 5020)	10,500 ± 5% (9975 to 11,025)

## Normal Force Calibration (for All Transducers)

Normal Force Calibration ensures that the transducer is properly measuring normal force. The calibration involves hanging a precision weight on the calibration tool, which is mounted on the transducer during calibration. The applied normal force is the amount of weight applied to the calibration tool. For example, hanging a 1000-gram weight applies a normal force of 1000 gmf.

### Normal Force Calibration Procedure

1. If calibrating an FRT, ensure that the transducer is set to High Range before proceeding. To set the transducer to High Range do the following:
  - a. Access the *Set Transducer Characteristics* form (Figure 5-7) by selecting the *Transducer* option from the *Service* function of the Utilities pull down menu.
  - b. Using the "Transducer Selected" menu, select the high range transducer then click "Ok"
2. Turn on the motor.
3. Raise the stage to maximum height and remove any test tools.
4. Install the calibration tool as shown in Figure 5-2 (the pulley need not be installed).
5. Using the *Set Gap/Instrument Control* function in Orchestrator, zero the normal force on the motor using the "Offset Normal Force to Zero" buttons.
6. Access the *Transducer Characteristics* form using the Orchestrator function *Calibrate Instrument* under the Utilities pull-down menu.
7. Select the "XducerCal" button. Establish a zero torque reference value by selecting the "Zero" button. Wait about 30 seconds, during which time the instrument takes several readings to establish a zero torque reference. When zeroing is completed, the *Transducer Calibration* form is displayed (Figure 5-4).

If, after selecting the "Zero" button, the NORMAL FORCE value displayed is either very high (such as 1E+5) or exactly zero, refer to the Troubleshooting Guide. The normal force should be less than 0.1% of the full scale normal force.

**NOTE:** Do not hang any weights until after at least one (1) zero reading has been taken.

8. Hang the weight specified in Table 5-4 (depending on the transducer in use) from the hook on the bottom of the calibration tool (Figure 5-6). Ensure that the weight is free to hang without obstruction, and that the weight is steady (not swinging from side-to-side).
9. Select the "Normal Cal" button. The Normal Calibration form is displayed.
10. Enter the APPLIED NORMAL FORCE specified in Table 5-4, depending on the transducer in use.

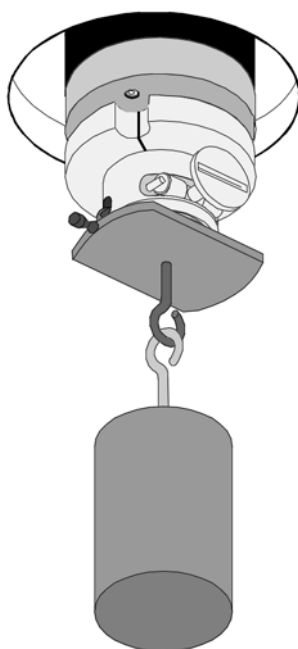


Figure 5-6. Applying Normal Force Using the Calibration Weight.

11. Select the "Calibrate Now" button. When calibration is complete, the *Transducer Calibration* form is displayed again.
12. Verify that the **Normal** value displayed on the *Transducer Calibration* Form (Figure 5-4) is within the limits for the NORMAL FORCE VALUE shown in Table 5-4, depending on the transducer in use. Verify that the **Current Normal Cal** value displayed in the *Transducer Calibration* form is within the limits for the CALIBRATED FULL SCALE VALUE shown in Table 5-4.

Table 5-4. Normal Force Calibration Weights, Applied Normal Forces, and Calibrated Full Scale Values

TRANSDUCER	WEIGHT	APPLIED NORMAL FORCE	NORMAL FORCE VALUE	CALIBRATED FULL SCALE VALUE
2K FRTN1 2K FRTN1E 1K FRTN1	1000 grams (p/n 613-01222)	1000 gmf	1000 ± 2 gmf (998 to 1002)	2100 gmf ± 5% (1995 to 2205)
100 FRTN1 200 FRTN1	100 grams (p/n 613-02060)	100 gmf	100 ± 0.1 gmf (99.9 to 100.1)	105 gmf ± 5% (99.75 to 110.25)
2K STD 10K STD	1000 grams (p/n 613-01222)	1000 gmf	1000 ± 5 gmf (995 to 1005)	1575 gmf ± 5% (1496.25 to 1653.75)

13. Select the "ACCEPT" button. Control returns to the *Transducer Characteristics* form.
14. Verify that the **Normal Calibration Value** now displayed in the form (the high range value if the transducer is an FRT) is the same as the **Current Normal Cal** value just displayed in the *Transducer Calibration* form. Press "Ok".
15. If the transducer in use is an FRT, remain in the *Transducer Setup* form and perform the following step. If the transducer is a STD transducer, go to step 16.
  - a. Access the *Set Transducer Characteristics* form (Figure 5-7) by selecting the *Transducer* option from the *Service* function of the *Utilities* pull down menu.
  - b. Copy the high range transducer **Normal Calibration Value** into the column containing the transducer settings for the low range transducer.
16. Click Ok.

This concludes the Normal Force Calibration. Remove the weight and calibration hook from the transducer, and store both in the calibration kit.

Transducer	1	2	3	4
Transducer Description	20GM FRT	1K FRT		
Maximum Torque [g·cm]	20.0	1000.0	0.0	0.0
Minimum Torque [g·cm]	0.004	1.0	0.0	0.0
Torque Calibration Value	20.42	1021.0	0.0	0.0
Torque Compliance	6.50e-06	6.50e-06	0.0	0.0
Inertia Constant [g·cm <sup>2</sup> ]	516.0	516.0	0.0	0.0
Phase Adjust Constant [rad·s/rad]	1.08e-04	1.08e-04	0.0	0.0
Maximum Normal Force [g]	2000.0	2000.0	0.0	0.0
Minimum Normal Force [g]	2.0	2.0	0.0	0.0
Normal Calibration Value	2101.6	2101.6	0.0	0.0
Normal Compliance [um/kg]	0.0	0.0	0.0	0.0
Phase Offset [rad]	0.0	0.0	0.0	0.0

Figure 5-7. Set Transducer Characteristics Form

Shaded windows show input cells for Torque and Normal Calibration values.

# Phase Angle Check

## Principle

The phase angle correction compensates for possible phase shifts that may be added to torque and strain due to A/D filtering by the instrument electronics. It is a function of the electronics and not the Motor or Transducer. Once the correction is determined, it should remain constant for the life of the system. The phase angle correction is computed, (and entered into Orchestrator) at the factory before the instrument is shipped, and should not have to be adjusted under normal operating conditions.

It is recommended that the phase angle be checked periodically using the following procedures to ensure that the entire system is functioning properly. If the values obtained from this test are abnormal, please contact Technical Services for further assistance.

To check the phase angle using the 2K FRTN1, 2K FRTN1E, 2K STD, or 10K STD Transducer, a Dynamic Frequency Sweep is run on a steel sample that is loaded into a torsion rectangular tool. Recall that a purely elastic sample has a phase angle of zero degrees. The phase angle of steel is near zero.

If using any of the 100 FRTN1, 200 FRTN1, 100 FRT or 200 FRT transducers, a Dynamic Frequency Sweep is run on a 1000 cP Newtonian fluid (calibration fluid) that is loaded onto a parallel plate tool. Again, recall that a purely viscous sample has a phase angle of 90 degrees. The phase angle of the Newtonian fluid is near 90 degrees.

To check the phase angle for the 1K FRTN1 transducer either the steel sample or oil sample may be used.

## Procedure for 1K FRTN1, 2K FRTN1, 2K FRTN1E, 2K STD, and 10K STD Transducers

1. Turn on the motor and ensure that it is in dynamic mode.
2. If the transducer is a 2K FRTN1, 2K FRTN1E, or 1K FRTN1, ensure that the transducer is in high range (use the Orchestrator function Utilities/Service/Transducer).
3. Install the Torsion Rectangular tool (see Chapter 4).
4. Load the calibration steel sample (part number 400-02589) that is supplied with the tool (see Chapter 4 for details).
5. Conduct a Dynamic Frequency Sweep using the following parameters:

FREQUENCY	0.1 to 100 rad/sec log sweep
STRAIN	0.02 %
TEMPERATURE	Current ambient temperature

Ensure that the phase angle,  $\delta$ , is  $\pm 0.25^\circ$  throughout the frequency range. If the values obtained from this test are outside this range, please contact Technical Services for further assistance.

## Procedure for 100 FRTN1, 200 FRTN1, 100 FRT, 200 FRT and 1K FRTN1

1. Turn on the motor and ensure that it is in dynamic mode.
2. In Orchestrator enter the *Edit/Start Instrument Test* function. Select Parallel Plate Geometry and then press the "Edit Geometry" button. Select the "Options" button and enter a fluid density of 1 g/cm<sup>3</sup> in the displayed form.
3. Ensure that the transducer range is in **high** range (use the Orchestrator function Utilities/Service/Transducer). When checking the 1K FRTN1 only, set the transducer to the **low** (20 g•cm) range.
4. Install the 50 mm parallel plate test tool(see Chapter 4).
5. Load the 1000 cP Newtonian Calibration fluid (part number 700-01016) with a gap of 1 millimeter.
6. Conduct a Dynamic Frequency Sweep using the following parameters:

FREQUENCY	0.1 to 100 rad/sec log sweep
STRAIN	100 %
TEMPERATURE	Current ambient temperature

Ensure that the phase angle,  $\delta$ , is between 87° and 92° throughout the frequency range. If the values obtained from this test are outside this range, please contact Technical Services for further assistance.

This concludes the phase angle check.

# Strain Calibration Check

Strain Calibration Check allows you to determine if actual strain (the angular deflection of the motor) corresponds to a commanded strain. In general, this procedure involves selecting the parallel plate geometry, then modifying the tool dimensions to achieve a strain constant of 1. This action results in commanded strain being identical to actual motor deflection, i.e., if you command 25% strain, the motor should deflect  $\pm 0.25$  radians from dynamic zero position.

While running a Dynamic Time Sweep, a calibration pointer attached to the motor allows you to visually examine motor position in relation to calibration marks etched onto the motor cover. The calibration marks are graduated in increments of 0.1, 0.25, and 0.5 radians from either side of dynamic zero position. Additionally, you can check the STRAIN reported by the Orchestrator online parameter display, which should indicate 25%.

## Procedure

1. Remove any test tools.
2. Turn on the motor and ensure that it is in dynamic mode.
3. Install the pointer onto the motor as shown in Figure 5-8.
4. Use the Strain Offset to align the pointer with dynamic zero position (Figure 5-9).
5. Select the parallel plate geometry as the current geometry, and enter the following tool dimensions:

DIAMETER	2 mm
GAP	1 mm

6. Set up a Dynamic Time Sweep with the following conditions:

FREQUENCY	0.3 rad/sec
STRAIN	25 %
TEMPERATURE	Current ambient temperature
TOTAL TIME	1000 sec
TIME PER MEASUREMENT	10 sec

7. Run the Dynamic Time Sweep.
8. Ensure that the following conditions exist:
  - a. The STRAIN value reported is between 24.90 and 25.10.
  - b. The pointer deflects  $\pm 0.25$  radians, as shown in Figure 5-9.

**If either condition is not met, contact TA Instruments Service group for further assistance.**

9. Stop the Dynamic Time Sweep.

This concludes the Strain Calibration Check.



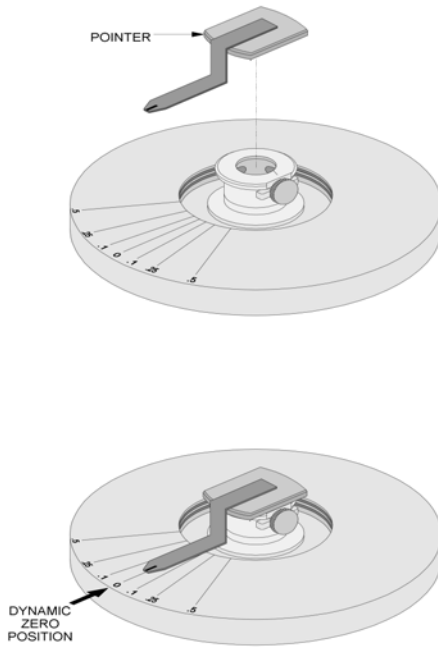


Figure 5-8. Installation of Pointer for Strain Calibration Check

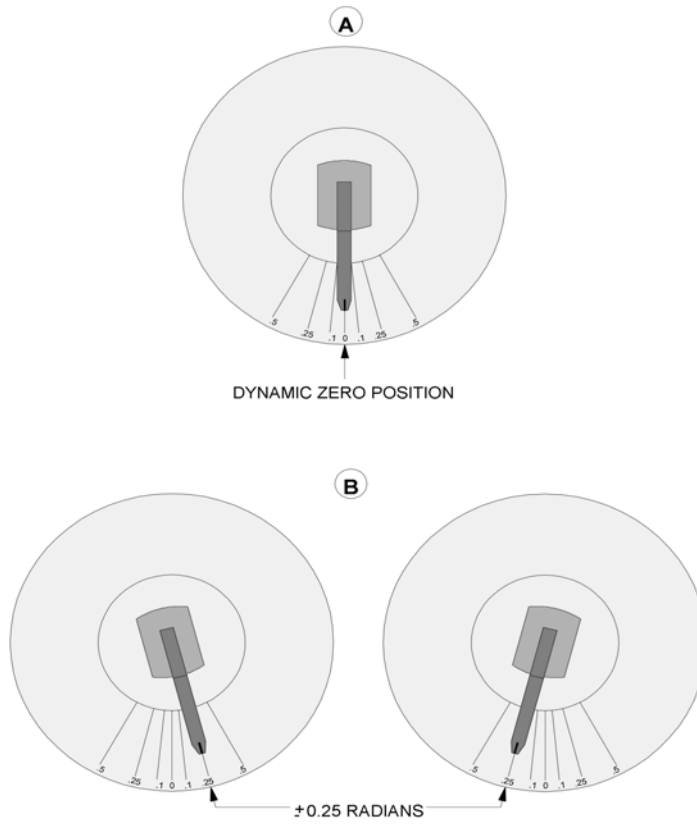


Figure 5-9. Strain Calibration Check with Pointer

## System Check Using PDMS

Included in the calibration kit (supplied with the instrument) is a jar of PDMS. PDMS (polydimethyl siloxane) is a rheological reference material that is used to verify the correct operation of the test station. A PDMS test should be run periodically to ensure proper operation of the instrument. It should also be run as a preliminary diagnostic any time there is a question regarding instrument performance.

### Procedure

1. Turn on the motor and ensure that it is in dynamic mode.
2. Ensure that the transducer range is in high range (use the Orchestrator function Utilities/Service/Transducer).
3. Install the 25-mm parallel plate test tool (see Chapter 4).
4. Load the PDMS (part number 700-01011) using a gap of 2 millimeter.
5. Conduct a Dynamic Frequency Sweep using the following parameters:

STRAIN	5 % (for 100 or 200 FRT/FRTN1 use 1%)
TEMPERATURE	30° C
SWEEP MODE	Log
INITIAL FREQUENCY	0.1 rad/s
FINAL FREQUENCY	100 rad/s
POINTS/DECADE	5

6. Set up the plot to show  $G'$ ,  $G''$ , viscosity, and phase angle. The displayed data should look similar to that shown in Figure 5-10.
7. At the completion of the test, select the  $G'/G''$  Crossover function from the Analysis pull down menu. The frequency, and value, where  $G'$  and  $G''$  cross (crossover point) is computed and displayed. Compare the computed frequency and  $G'/G''$  crossover to the values labeled on the PDMS jar. Please note that on the PDMS jar, crossover frequency (in rad/s) is labeled  $W_c$ , and the crossover value is labeled  $G_c$  (no exponent is shown). The computed crossover point should be within the error limits provided on the jar. Press the "Stamp" button to place the computed crossover data on the plot.

This concludes the PDMS test.

### Dynamic Frequency Sweep of PDMS

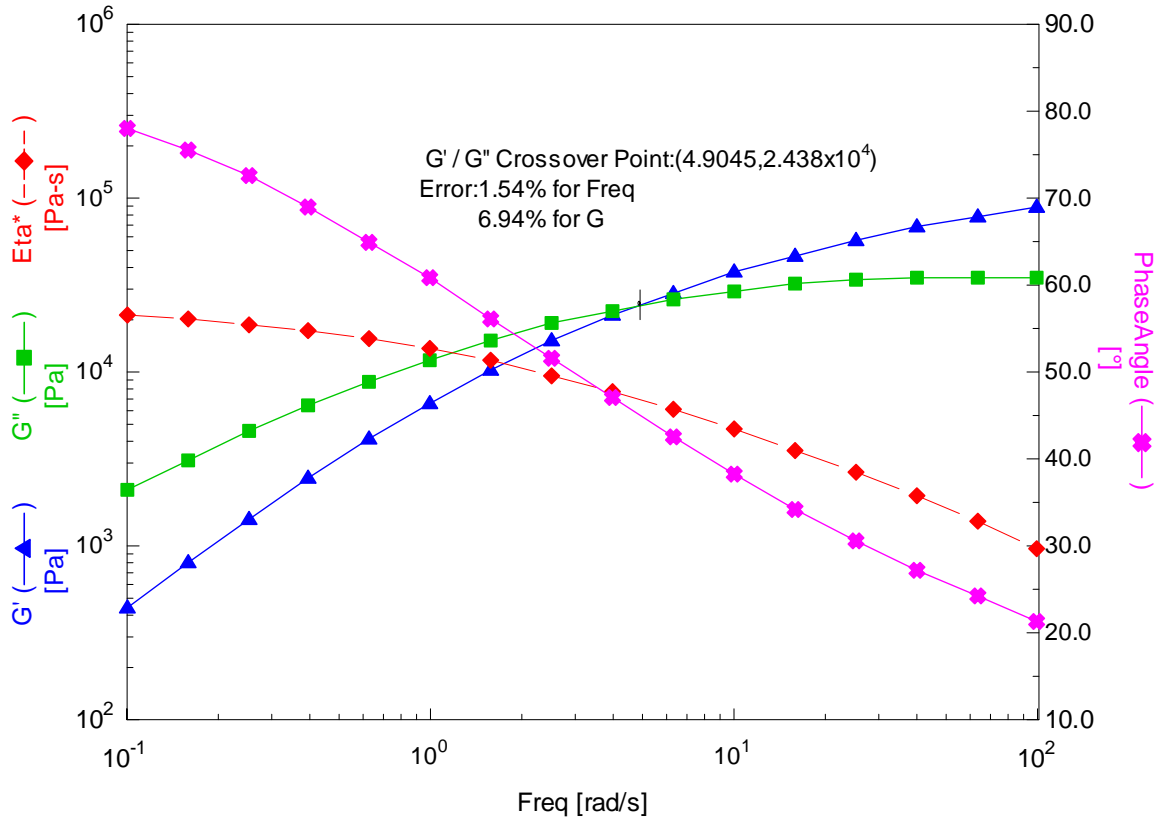


Figure 5-10. Typical Results of PDMS Tests

# Temperature Calibration

The temperature of the ARES is monitored by a Platinum Resistance Thermometer, (PRT) which is automatically calibrated upon power up. This calibration is accomplished by switching in precision resistors in place of the PRT and using the known resistance to adjust the offset and gain of the electronics. However, some errors can still exist due to PRT errors, or temperature gradients that exist within the oven, which cause the temperature at the PRT to truly be different than the temperature at the sample. Although these errors are generally small, they can lead to discrepancies between the temperature measured by the PRT and the actual temperature of the sample at any given time.

For studies that require the most exacting temperature accuracy, it can be helpful to input a temperature calibration table to account for differences in measured temperature versus actual sample temperature. However, keep in mind that performing the calibration incorrectly could actually make the errors worse than doing nothing. Of prime consideration is providing a temperature reference that is of sufficient accuracy to be used as a calibration reference. One option is to mount an accurate thermometer in place of the sample and compare its output to that of the PRT at several temperature steps. Another method used is to run a temperature ramp study on a material with well defined and well known thermal transitions (such as the glass transition point) and using the reported values to adjust the PRT temperatures.

## *Temperature Calibration using Orchestrator™*

While temperature calibration is *not* handled under the standard instrument calibration menu screens, or procedures, we provide the following option in Orchestrator for adjusting the ARES for temperature error effects. Please note that this option is only available for ARES firmware version 5.xx and above.

Obtain a table of calibration temperatures relative to PRT temperatures using either a reference thermometer or known samples transition points as described above. If using material transition points, more than one material should be used for greater precision across a broad range of temperatures.

Under the Utilities pull down menu select the *Instrument Configuration* function from the *Service* function table. Select TEMPERATURE CONTROL (Figure 5-11). Near the bottom of the form, click "Adjustable" for the Temperature Calibration Table. A table will be displayed with up to 20 windows to input the calibration values. Enter the measured and theoretical values for each calibration points obtained, leaving the remaining windows unaltered. The ARES will now adjust temperatures measured by the PRT according to the calibration table, linearly interpolating between table values.

It is recommended that you enter the same commanded and calibrated values for room temperature so that there is no offset at room temperature.

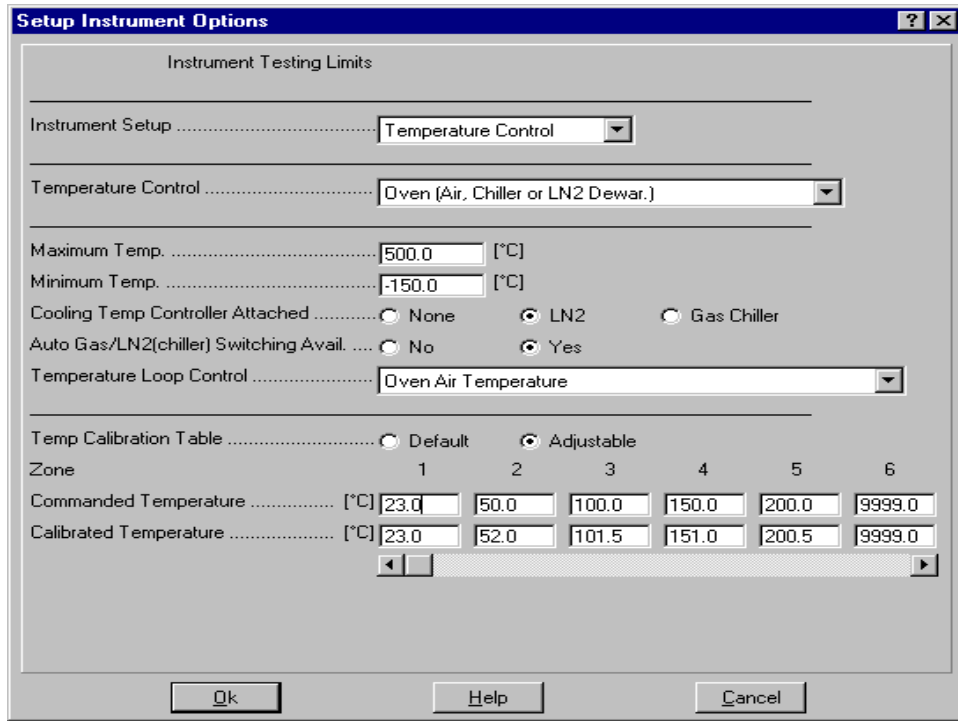


Figure 5-11. Setup Instrument Temperature Options Showing the Adjustable Temperature Calibration Table



---

---

# Chapter 6

## Maintenance

### General Information

This chapter contains the following information:

- **Routine Maintenance:** Routine maintenance consists of tasks that we recommend you perform on a periodic basis.
- **Special Maintenance:** Special maintenance tasks can be performed only by qualified electronic technicians.
- **Troubleshooting Guide:** A troubleshooting guide is supplied to assist you in diagnosing selected problems.

### Routine Maintenance

#### *Cable and Hose Inspection*

Damage to the AC power cords can cause a safety hazard. Periodically inspect these items as follows.

#### AC Power Cords

Remove AC power to the instrument as follows:

1. Push the Main Power Switch to the OFF (O) position.
2. Remove the POWER IN plug from the AC main source.
3. Inspect all the cords for frayed insulation or exposed bare copper wire, especially in the immediate vicinity of the plugs on either end. If any damage is found, notify TA Instruments Technical Service.

Apply AC power to the instrument as follows:

1. Install the POWER IN plug in the AC main (line voltage source).
2. Push the Main Power Switch to the ON (I) position.

#### Air Hoses

Remove AC power to the instrument as follows:

1. Push the Main Power Switch to the OFF (O) position.
2. Remove the POWER IN plug from the AC main (line voltage source).
3. Lock the Transducer and Motor bearings.
4. Remove Air supply to instrument.

Inspect the air hoses for cracks and other damage that could result in leaks, especially in the vicinity of the bend radii. If any damage is found, notify TA Instruments for service.

There are also internal filters inside the test station that should be inspected and serviced on an approximately 6 month to 1-year basis. Only qualified service personal should perform this maintenance.

If no leaks are found, apply AC power to the instrument as follows:

1. Establish airflow through the air dryer, but do not connect the air output to the test station. Allow the air to purge for 3 to 5 minutes.
2. Connect the air supply to the test station.
3. Unlock the transducer and motor bearings.
4. Install the POWER IN plug in the AC main (line voltage source).
5. Push the Main Power Switch to the ON (I) position.

Verify that the air pressure to the motor, transducer, and oven are correct. Note: to check the oven air pressure the oven must be all the way to the right and closed.

## Air Dryer

Inspect and service the air dryer according to its manual. This should include draining and cleaning the pre-filters. Replace them if necessary.

## *Cleaning the Instrument*

If the exterior plastic or metallic surfaces of the instrument require cleaning, use only a solution consisting of a non-abrasive household dish detergent and water.

Clean as follows:

1. Remove AC power to the instrument as follows:
  - a. Push the Main Power Switch to the OFF (O) position.
  - b. Remove the POWER IN plug from the AC main (line voltage source).
2. Apply some cleaning solution onto a cotton cloth, then wring out the cloth to discharge excess water; the cloth must be damp, but not wet.
3. While ensuring that excess fluid from the cloth does not enter any crevice of the instrument, use the cloth to gently clean the desired external surfaces.
4. Ensure that all surfaces of the instrument are dry.
5. Apply AC power to the instrument as follows:
  - a. Install the POWER IN plug in the AC main (line voltage source).
  - b. Push the Main Power Switch to the ON (I) position.



## *Lifting and Carrying the Instrument*

### Test Station

The Test Station weighs 275 pounds (125 kilograms). It can be safely lifted and carried only by a fork lift that is rated to carry such weight. However, since there is no surface that offers adequate contact points for a fork lift, the Test Station is designed to be lifted only when it is mounted onto a pallet (this is the shipping configuration) by placing the forks in the pallet.

The handles on the side of the test station can be used to slide the test station on the workbench surface. You may need to apply some lift while sliding the instrument, but do not attempt to completely lift and carry the Test Station by the handles, as balancing the instrument is difficult. Be careful not to damage the feet on the bottom of the Test Station.

During installation, the Test Station will be removed from the pallet and placed on the workbench. Retain the pallet for future use in case the Test Station requires moving. To place the Test Station back on the pallet, use a forklift to position the pallet flush with the workbench top and "slide" the Test Station onto the pallet.



**WARNING: Do not attempt to lift or carry the Test Station by hand. Use a fork lift that is rated to carry the weight of the Test Station. Attempting to lift or carry the Test Station by hand can result in serious personal injury or damage to the Test Station.**

---

# Special Maintenance

## *Service and Repair of the Test Station*

Other than the routine maintenance listed in the previous section, there are no other repairs or service that you, as the customer, can perform. Contact TA Instruments regarding service or repairs, as well as the availability of service contracts and plans.

## *Diagnostic LEDs*

Each removable circuit board on the Test Station Mother Board is equipped with a bank of diagnostic LEDs (light-emitting diodes) that indicate the operational status of key electrical signals. Figure 6-1 shows the location of the LEDs, which can be viewed by opening the access door. The electronics are protected by a metal shield that allows inspection of the LEDs during basic troubleshooting.

The electrical signal monitored by each LED is labeled. Except as noted in Figure 6-1, an illuminated LED indicates proper operation.

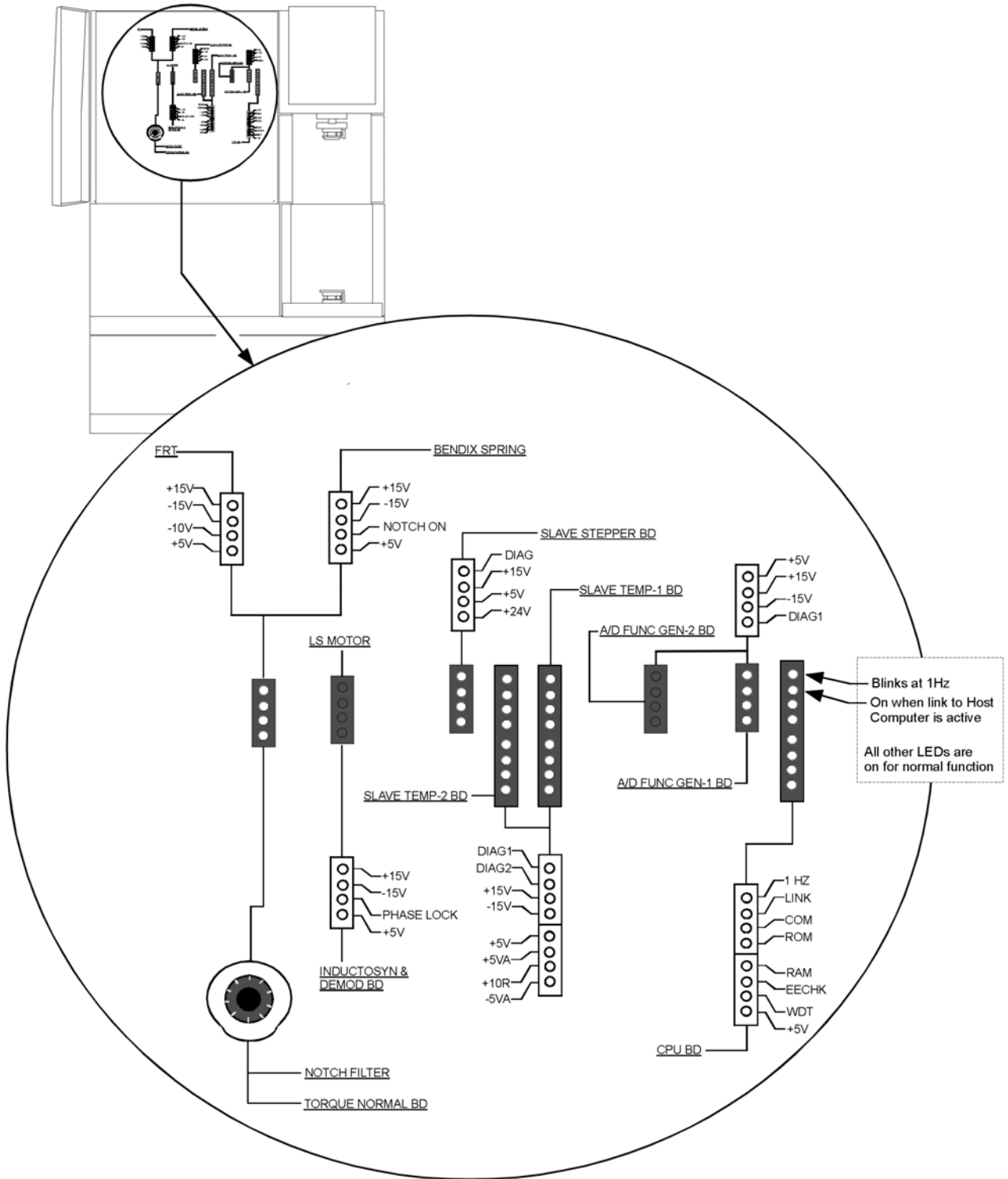


Figure 6-1. Location of Diagnostic LEDs

# Troubleshooting Guide

Table 6-1 and Table 6-2 list some problems that may arise during instrument calibration and operation, and offers the corresponding corrective actions. Each corrective action is listed by number. Each action constitutes a single troubleshooting operation. For example, first try corrective action 1. If the problem persists, try 2, and so on until you reach *Call Technical Service*.

Before calling technical service have the following information ready:

- The instrument model and serial number
- The software version you are running.
- Any troubleshooting steps you followed to diagnose the problem.

## Calibration

Table 6-1. Calibration Troubleshooting Guide

PROBLEM	CORRECTIVE ACTIONS
<b>NORMAL FORCE CALIBRATION</b>	
<p>During either Torque or Normal Force Calibration, the first value of force that is displayed is either very high (such as 1E+5) or exactly zero.</p>	<ol style="list-style-type: none"> <li>1) Exit the calibration operation and enter the TRANSDUCER SETUP (UTILITIES: SERVICE: TRANSDUCER). The TORQUE (or NORMAL) CALIBRATION VALUE is nominally about 5% higher than the maximum force that is measurable by the transducer in use. This value should not be zero, nor should it be a very large number such as 1E+5. If it is, enter the correct full scale value for the transducer in use (see <i>Instrument Specifications</i>), then repeat the calibration.</li> <li>2) Exit the calibration operation and cycle the instrument main power (turn off, then on). Repeat the calibration.</li> <li>3) Call Technical Service</li> </ol>
<b>PHASE ANGLE CALIBRATION</b>	
<p>During Phase Angle Calibration with a steel sample, the phase angle is not within the limits specified in the calibration procedure.</p>	<ol style="list-style-type: none"> <li>1) Repeat the calibration (only once).</li> <li>2) Check that entered phase correction and offset are reasonable.</li> <li>3) Ensure sample is loaded correctly.</li> <li>4) Verify static force is correct and in proper direction.</li> <li>5) Remove the sample, exit the calibration operation, then cycle the instrument main power (turn off, then on). Re-load the sample and repeat the calibration.</li> <li>6) Call Technical Service</li> </ol>

# Operation

Table 6-2. Instrument Operation Troubleshooting Guide

PROBLEM	CORRECTIVE ACTIONS
<b>MOTOR</b>	
Motor does not engage or respond to command.	<ol style="list-style-type: none"> <li>1) Ensure that the motor is on.</li> <li>2) Ensure that the instrument is not in an OVERLOAD condition. OVERLOAD is indicated by an online indicator. If an OVERLOAD is indicated, reset the test using the function END TEST/RESET.</li> <li>3) <i>LS Motor Only</i>: Ensure that motor air pressure is 60 psi.</li> <li>4) Call Technical Service.</li> </ol>
Motor Displacement is incorrect.	<ol style="list-style-type: none"> <li>1) Ensure that correct geometry is entered.</li> <li>2) Ensure that the desired strain is entered.</li> <li>3) Verify that the diagnostic LEDs are lit.</li> <li>4) Call Technical Service.</li> </ol>
Motor oscillates with an accompanying high-pitched, audible noise when running a test.	<ol style="list-style-type: none"> <li>1) Ensure sample is not too stiff.</li> <li>2) Call Technical Service.</li> </ol>
<b>OVEN AND LN2 CONTROLLER</b>	
Can not turn on the oven.	<ol style="list-style-type: none"> <li>1) If air is being used as an input to the heaters, ensure that the Orchestrator AIR LOW indicator is not on. If it is, the air supply to the oven has been interrupted. Restore air flow.</li> <li>2) If using the LN2 Controller, ensure that:               <ul style="list-style-type: none"> <li>The LN2 supply is adequate and the valve is open.</li> <li>The LN2 READY indicator is on.</li> <li>The LN2 FAULT indicator is off (if on, go to 7).</li> </ul> </li> <li>3) Ensure that the oven door is closed and the OVEN OPEN indicator is not on.</li> <li>4) Ensure that the reported temperature is not constantly above 650 °C. If it is, there is an open in the PRT electrical circuit. Go to 8.</li> <li>5) Ensure that the SET USER TEMPERATURE LIMIT is set to a reasonable value.</li> <li>6) Ensure that the instrument MAXIMUM TEMPERATURE is set to a reasonable value.</li> <li>7) An oven or heater fuse may be open. Check and replace.</li> <li>8) Call Technical Service.</li> </ol>

Table 6-2. Instrument Operation Troubleshooting Guide (Continued)

PROBLEM	CORRECTIVE ACTIONS
Oven can be turned on, but does not heat.	<ol style="list-style-type: none"> <li>1) Ensure that Orchestrator online indicators do not indicate a problem; if so go to 7.</li> <li>2) Ensure that the commanded temperature is correct and reasonable; if not, command a suitable temperature.</li> <li>3) Ensure that the SET USER TEMPERATURE LIMIT is set to a reasonable value; if not, set accordingly.</li> <li>4) Ensure that the temperature reported by Orchestrator is close to actual temperature. If the temperature is 650°C, the one or both of the PRTs are open.</li> <li>5) Visually inspect PRTs for damage. If damaged, go to 7.</li> <li>6) An oven fuse may be open.</li> <li>7) Call Technical Service.</li> </ol>
Oven can be turned on, but is not correctly heating.	<ol style="list-style-type: none"> <li>1) Ensure that the temperature reported by Orchestrator is close to actual temperature; if not, change temperature control to Mode 3. If reported temperature is now correct, then Tool PRT or upper oven PRT control loop is malfunctioning. If reported temperature is incorrect then lower oven PRT control loop is malfunctioning.</li> <li>2) Visually inspect PRTs for damage. If damaged, go to 4.</li> <li>3) An oven fuse may be open.</li> <li>4) Call Technical Service.</li> </ol>
No LN2 READY indication (Orchestrator).	<ol style="list-style-type: none"> <li>1) Ensure that LN<sub>2</sub> supply pressure is adequate (<i>i.e.</i>, 20-30 psi); if not, check supply lines.</li> <li>2) Ensure that LN<sub>2</sub> filter is not clogged; if so, go to 5.</li> <li>3) Ensure that, when the LN2 Controller is on, vent gas exits from the muffler on the LN2 Controller; if not, go to 5.</li> <li>4) Ensure that there is no leaking around dewar.</li> <li>5) Call Technical Service.</li> </ol>

Table 6-2. Instrument Operation Troubleshooting Guide (Continued)

PROBLEM	CORRECTIVE ACTIONS
<b>TRANSDUCER</b>	
Torque or Normal Force either does not respond, or is beyond full-scale when no force is applied.	<p><i>Applies to FRT transducers only:</i></p> <ol style="list-style-type: none"> <li>1) Ensure that the correct air pressure is supplied to the transducer in use (see <i>instrument specifications</i>); if pressure is incorrect, go to 3.</li> <li>2) Ensure that the transducer is unlocked.</li> <li>3) Call Technical Service.</li> </ol>
<b>STAGE</b>	
Stage does not move.	<p>NOTE: Manual AutoTension is activated by selecting the HOLD button (see Orchestrator Online Help for details).</p> <ol style="list-style-type: none"> <li>1) Make sure the stage is not at its limit of its travel.</li> <li>2) Ensure that there is not a force overload condition.</li> <li>3) Ensure that manual AutoTension functions correctly; if not, go to 5.</li> <li>4) Ensure that manual AutoTension is not on when trying to move stage using the Stage Control; if it is on, turn it off.</li> <li>5) Ensure it is activated in firmware.</li> <li>6) Cycle the instrument main power (turn off, then on).</li> <li>7) Ensure that the oven is all the way to the left or all the way to the right.</li> <li>8) Call Technical Service.</li> </ol>
Stage moves in only one direction when AutoTension is not in use.	<ol style="list-style-type: none"> <li>1) Ensure that a Normal Force Overload condition does not exist; if it does, determine the cause.</li> <li>2) Ensure that the stage is not at the limit of its travel; if it is, adjust accordingly.</li> <li>3) Cycle the instrument main power (turn off, then on).</li> <li>4) Call Technical Service.</li> </ol>
Stage moves in only one direction when AutoTension is in use.	<ol style="list-style-type: none"> <li>1) During sample loading, ensure that the stage is not at the limit of its travel; if it is, adjust accordingly.</li> <li>2) Ensure that the AutoTension Window is set to a reasonable value; if not, re-set.</li> <li>3) Call Technical Service.</li> </ol>

Table 6-2. Instrument Operation Troubleshooting Guide (Continued)

PROBLEM	CORRECTIVE ACTIONS
<b>RUNNING A TEST AND COLLECTING DATA</b>	
Erratic data points or missing data.	<ol style="list-style-type: none"> <li>1) <i>For FRT Transducers Only:</i> Ensure that the correct air pressure is supplied to the transducer in use (see <i>instrument specifications</i>); if pressure is incorrect, go to 7.</li> <li>2) Ensure that AutoTension is functioning correctly; if not, go to 8.</li> <li>3) Ensure that the oven is not in contact with the upper and lower test tools; if so, go to 8.</li> <li>4) Ensure that the sample is loaded correctly.</li> <li>5) Cycle the instrument main power (turn off, then on).</li> <li>6) Ensure on-line indicator shows ON TEMP.</li> <li>7) Run confidence check with steel shim.</li> <li>8) Perform a torque calibration.</li> <li>9) Perform a strain check.</li> <li>10) Check force level for overload condition.</li> <li>11) Check that pretension is correct.</li> <li>12) Make sure the sample stiffness/dimensions are reasonable for the test tool.</li> <li>13) Call Technical Service.</li> </ol>



## A

### AC Power

- main power switch, 47
- specifications, 24

Air. *See* Gas, input options

Air Dryer, 29, 50, 51, 232

Air Quality, 50

Analog Data Input, 124, 143

Arbitrary Waveshape Test, 126

AutoStrain, 144

AutoTension, 141

## B

### Bearing Locks

- FRT with normal force, 37
- FRT without normal force, 41
- low shear (LS) motor, 42
- standard motors, 41
- Standard Transducer, 37

Boltzmann Superposition Principle, 111

## C

### Calibration

- normal force, 219
- phase angle check, 222
- strain check, 224
- temperature, 228
- torque, 214

Calibration Intervals, 212

calibration procedures, 211

## Chiller

- description, 55
- diagram, 56
- electrical specifications, 25
- physical specifications, 24

circulator, 23, 26, 44, 63, 66, 73, 85

Cleaning the Instrument, 232

COM Port, 44

Complex Modulus ( $E^*$ ), 92

## Compliance

- definition, 148
- determination of operational range, 149

## Cone and Plate (test fixture)

- constants, 163
- fixture installation, 165
- general information, 163
- operating range, 164
- sample loading, 166

Constant Stress Test, 136

Correlation Delay, 145

## Couette (test fixture)

- constants, 181
- fixture installation, 183
- general information, 181
- operating range, 181
- sample loading, 183
- vane tool, 184

Creep, 125

## D

Delay Before Test, 140

Dewar Flask, 57

Diagnostic LEDs, 234

Dilatancy, 92

## Double Wall Couette (test fixture)

- constants, 186
- fixture installation, 188, 192

- general information, 186
- operating range, 187
- sample loading, 189, 192

Dynamic Measurement Formulas, 207

Dynamic Single Point Measurement, 100

Dynamic Strain Sweep, 98, 106

Dynamic Temperature Ramp, 108, 109

Dynamic Temperature Step, 104

Dynamic Time Sweep, 98, 101

## E

$E'$ . *See* Elastic Modulus

$E''$ . *See* Viscous Modulus

$E^*$ . *See* Complex Modulus

Elastic Modulus ( $E'$ ), 92

Elastic Stress ( $\tau'$ ), 92

Elasticity, 91

Elastomers, 147

Emulsions, 147

Environmental Control System

- configuring, 60

- description, 53

- principles of operation, 23

- specifications, 26

Errors

- discrepancies in sample geometry, 150

- temperature variations, 151

- testing outside linear region, 150

Extensional Stress, 195

Extensional Viscosity, 195

Extensional Viscosity Fixture, 195

- Extensional Viscosity Fixture (EVF), 195
  - description, 195
  - hot compression molding samples, 197
  - installing fixture, 198
  - instrument preparation, 199
  - plotting data, 202
  - running experiments, 201
  - sample loading, 201
  - sample preparation, 196
  - sample support, 196
  - schematics, 196
  - test setup, 199

## F

- Fluid Bath
  - circulator connections, 84
  - description, 82
  - inet line clamp, 85
  - installation, 82
  - lower fixture, 84
  - operating specifications, 26
  - operation, 89

- Fluid Bath 2
  - circulator connections, 66
  - description, 63
  - installation, 63
  - lower fixtures, 67
  - operation, 70
  - PRT installation, 66

- Force Gap test, 133

- Force Gap Test, 133

- Frequency Sweep, 98, 102, 103

- Frequency/Temperature Sweep, 98, 114

## G

- Gap
  - automatic zero, 156
  - enabling the gap control panel, 156
  - manual zero, 155
  - max force allowed option, 157
  - read text fixture gap, 157
  - set gap/instrument control function, 34
  - setting, general, 155

## Gas

- input options, 49
- pressure specifications, 49

## H

### Helical Ribbon Fixture, 204

- characteristics, 204
- couette analogy, 205
- description, 204
- environmental systems, 204
- operating range, 206
- options, 204
- sample loading, 206
- tool installation, 206

### Hencky Rate, 195

### Hencky Strain, 195

### Hencky Strain Rate, 124

### high voltage warning, 211

### Hookean Region, 91

### Hooke's Law, 91

### Host Computer, 31

### hot compression molding, 197

- for EVF, 197

### Humidity Cover, 80, 81

## I

### Instrument Control Panel, 32, 61

## L

### LCD Display, 36

### LEDs

- diagnostic, 234

### Linear Region, 91

### Linear Strain Sweep, 106

## LN<sub>2</sub> Controller

- description, 57
- location, 29
- physical specifications, 24

Logarithmic Strain Sweep, 106

Loss Modulus. *See* Viscous Modulus

## M

Main Power Switch, 47

### Maintenance

- cleaning, 232
- routine, 231
- special, 234

Manual Delay, 140

Modulus, 91

### Motor

- air pressure requirements, 49
- description, 31
- location, 30
- principles of operation, 22
- rapid shut off, 32
- specifications, 26
- turning on and off, 32

Multiple Extension Mode, 124

MultiWave Single Point, 111

MultiWave Temperature Ramp, 111

## N

Newton's Law, 91

## O

One Cycle Correlation, 145

### Oven

- description, 54
- gas selection, 58
- location, 30
- operating requirements, 59

- operation, 61
- rapid shutdown, 32
- signal connection to test station, 44
- temperature control, 54

Oven PRT, 61

## P

Parallel Plates (test fixture)

- constants, 159
- fixture installation, 161
- general information, 159
- operating range, 159
- sample loading, 161

Peltier

- description, 72
- humidity cover, 80
- installation, 74
- operating specifications, 26
- operation, 80
- PID loop setup, 79
- selecting operating range, 73

Phase Angle

- description, 92
- shift ( $\delta$ ), 92

Platinum Resistance Thermometer. *See* PRT

Pneumatics Panel

- connections, 50
- description, 49
- location, 43

Polymer Melts, 148

Power Panel

- connections, 47
- description, 46
- location, 43

PreTension. *See* AutoTension

PRT, 23, 54, 61, 228, 237

Pseudoplasticity, 92

## R

Regulatory Compliance, 10

Rim Shear, 124

## S

Sample PRT, 61

samples

loading on EVF, 201

offset loading on EVF, 201

preparing by hot compression molding, 197

Service and Repair, 234

Shear Rate, 91

Shear Stress, 91

Shear Thickening. *See* Dilatancy

Shear Thinning. *See* Pseudoplasticity

Signal Panel

connectors, 45

description, 43

general connections, 43

location, 43

Squeeze Flow, 124

Stage

description, 33

location, 30

Stage Control

manual, 33

rate adjustment, 35

software, 34

Steady and Transient Measurement Formulas, 208

Steady PreShear, 139

Steady Rate Sweep, 117

Steady Single Point, 115

Steady Step Rate Temperature Ramp, 134



Step Shear Rate, 120

Stiffness, 148

Storage Modulus. *See* Elastic Modulus

Strain, 91, 92, 94, 148

Strain-Controlled Steady Test Methods, 115

Strain-Controlled Transient Test Methods, 120

Stress, 91, 92, 94

Stress Ramp Test, 138

Stress Relaxation, 98, 122

Suspensions, 147

## T

Tan  $\delta$ , 93

Technical Support, 10

Temperature Calibration, 228

Tensile Force, 195

Tensile Strain, 91

Tensile Stress, 91

Test Fixtures

    general guidelines, 152, 158

    upper fixture installation, 152

Test Modes, 98

Test Station, 31

Thermal Expansion, 158

Thermoplastics, 147

Thermosets, 147

Thermosetting Resins, 148

Thin Films, 147

Thixotropic Loop, 129

Torque/Normal Relaxation, 131

Torsion Rectangular, New Design (test fixture)

constants, 167

fixture installation, 170

general information, 167

operating range, 168

sample loading, 171

Torsion Rectangular, Original Design (test fixture)

constants, 175

fixture installation, 178

general information, 175

operating range, 176

sample loading, 178

Transducer

description, 33

principles of operation, 22

Transient Step Strain, 122

Troubleshooting Guide, 236

## V

Vane Tool. *See* Couette

Viscoelastic, 91

Viscosity, 91

Viscous Modulus ( $E''$ ), 92

Viscous Stress ( $\tau''$ ), 92

# Appendix A

## Complex Modulus Limits

This appendix provides complex modulus limits for various tools.

Appendix Table A1-1. Complex Modulus Limits for Parallel Plate:  
2K FRTN1 and 2K FRTN1E Transducers.

PLATE DIAMETER (mm)	GAP (mm)	G* MAXIMUM (dynes/cm <sup>2</sup> )	G* MINIMUM (dynes/cm <sup>2</sup> )
25	2	4.45E+06	2.05E+02 (High range) 2.05E+00 (Low range)
	1	2.22E+06	1.02E+02 (High range) 1.02E+00 (Low range)
	0.5	1.11E+06	5.11E+01 (High range) 5.11E-01 (Low range)
50	2	2.78E+05	1.28E+01 (High range) 1.28E-01 (Low range)
	1	1.39E+05	6.39E+00 (High range) 6.39E-02 (Low range)
	0.5	6.95E+04	3.20E+00 (High range) 3.20E-02 (Low range)

Appendix Table A1-2. Complex Modulus Limits for Parallel Plate: STD Transducer.

PLATE DIAMETER (mm)	GAP (mm)	G* MAXIMUM (dynes/cm <sup>2</sup> )	G* MINIMUM (dynes/cm <sup>2</sup> )
25	2	1.97E+06	2.05E+02 (2K STD) 1.02E+03 (10K STD)
	1	9.84E+05	1.02E+02 (2K STD) 5.10E+2 (10K STD)
	0.5	4.92E+05	5.11E+01 (2K STD) 2.56E+02 (10K STD)
50	2	1.23E+05	1.28E+01 (2K STD) 6.40E+01 (10K STD)
	1	6.15E+04	6.39E+00 (2K STD) 4.09E+01 (10K STD)
	0.5	3.07E+04	3.20E+00 (2K STD) 1.60E+01 (10K STD)

Appendix Table A1-3. Complex Modulus Limits for Parallel Plate: 100 and 200 FRTN1 Transducers.

PLATE DIAMETER (mm)	GAP (mm)	G* MAXIMUM (dynes/cm <sup>2</sup> ) at Frequency $\omega$ (rad/sec)	G* MINIMUM (dynes/cm <sup>2</sup> )
25	1	9.84E+04 $\omega = 100$ 9.84E+05 $\omega = 10$ 5.11E+07 $\omega < 10$	2.05E+00 (High range, 100 FRTN1) 4.10E+00 (High range, 200 FRTN1) 2.05E-01 (Low range, 100 FRTN1) 4.10E-01 (Low range, 200 FRTN1)
	0.5	4.92E+04 $\omega = 100$ 4.92E+05 $\omega = 10$ 2.56E+07 $\omega < 10$	1.02E-00 (High range, 100 FRTN1) 2.04E+00 (High range, 200 FRTN1) 1.02E-01 (Low range, 100 FRTN1) 2.04E-01 (Low range, 200 FRTN1)
50	1	6.15E+03 $\omega = 100$ 6.15E+04 $\omega = 10$ 3.20E+06 $\omega < 10$	1.28E-01 (High range, 100 FRTN1) 2.66E-01 (High range, 200 FRTN1) 1.28E-02 (Low range, 100 FRTN1) 2.66E-02 (Low range, 200 FRTN1)
	0.5	3.07E+03 $\omega = 100$ 3.07E+04 $\omega = 10$ 1.60E+06 $\omega < 10$	6.39E-02 (High range, 100 FRTN1) 1.28E-01 (High range, 200 FRTN1) 6.39E-03 (Low range, 100 FRTN1) 1.28E-02 (Low range, 200 FRTN1)

Appendix Table A1-4. Complex Modulus Limits for Parallel Plate: 100 and 200 FRT Transducers.

PLATE DIAMETER (mm)	GAP (mm)	G* MAXIMUM (dynes/cm <sup>2</sup> ) at Frequency $\omega$ (rad/sec)	G* MINIMUM (dynes/cm <sup>2</sup> )
25	1	9.84E+04 $\omega = 100$ 9.84E+05 $\omega = 10$ 5.11E+07 $\omega < 10$	1.02E+00 (High range, 100 FRT) 2.04E+00 (High range, 200 FRT) 1.02E-01 (Low range, 100 FRT) 2.04E-01 (Low range, 200 FRT)
	0.5	4.92E+04 $\omega = 100$ 4.92E+05 $\omega = 10$ 2.56E+07 $\omega < 10$	5.11E-01 (High range, 100 FRT) 1.02E+00 (High range, 200 FRT) 5.11E-02 (Low range, 100 FRT) 1.02E-01 (Low range, 200 FRT)
50	1	6.15E+03 $\omega = 100$ 6.15E+04 $\omega = 10$ 3.20E+06 $\omega < 10$	6.39E-02 (High range, 100 FRT) 1.28E-01 (High range, 200 FRT) 6.39E-03 (Low range, 100 FRT) 1.28E-02 (Low range, 200 FRT)
	0.5	3.07E+03 $\omega = 100$ 3.07E+04 $\omega = 10$ 1.60E+06 $\omega < 10$	3.20E-02 (High range, 100 FRT) 6.40E-02 (High range, 200 FRT) 3.20E-03 (Low range, 100 FRT) 6.40E-03 (Low range, 200 FRT)

Appendix Table A1-5. Complex Modulus Limits for Cone and Plate:  
2K FRTN1 and 2K FRTN1E Transducers.

PLATE DIAMETER (mm)	CONE ANGLE (rad)	G* MAXIMUM (dynes/cm <sup>2</sup> )	G* MINIMUM (dynes/cm <sup>2</sup> )
25	0.1	2.08E+06	9.59E+01 (high range) 9.59E-01 (low range)
	0.02	4.17E+05	1.92E+01 (high range) 1.92E-01 (low range)
	0.04	8.34E+05	3.84E+01 (high range) 3.84E-01 (low range)
50	0.02	5.21E+04	2.40E+00 (high range) 2.40E-02 (low range)
	0.04	1.04E+05	4.79E+00 (high range) 4.79E-02 (low range)

Appendix Table A1-6. Complex Modulus Limits for Cone and Plate:  
2K and 10K Standard Transducer.

PLATE DIAMETER (mm)	CONE ANGLE (rad)	G* MAXIMUM (dynes/cm <sup>2</sup> )	G* MINIMUM (dynes/cm <sup>2</sup> )
25	0.1	9.22E+05	9.59E+01 (2K) 4.80E+02 (10K)
	0.02	1.84E+05	1.92E+01 (2K) 9.60E+01 (10K)
	0.04	3.69E+05	3.84E+01 (2K) 1.92E+02 (10K)
50	0.02	2.31E+04	2.40E+00 (2K) 1.20E+01 (10K)
	0.04	4.61E+04	4.79E+00 (2K) 2.40E+01 (10K)

Appendix Table A1-7. Complex Modulus Limits for Cone and Plate: FRTN1 Transducer.

PLATE DIAMETER (mm)	CONE ANGLE (rad)	G* MAXIMUM (dynes/cm <sup>2</sup> ) at Frequency $\omega$ (rad/sec)	G* MINIMUM (dynes/cm <sup>2</sup> )
25	0.1	9.22E+04 $\omega = 100$ 9.22E+05 $\omega = 10$ 4.79E+07 $\omega < 10$	1.92E+00 (High range, 100 FRTN1) 3.84E+00 (High range, 200 FRTN1) 1.92E-01 (Low range, 100 FRTN1) 3.84E-01 (Low range, 200 FRTN1)
	0.02	1.84E+04 $\omega = 100$ 1.84E+05 $\omega = 10$ 9.59E+06 $\omega < 10$	3.84E-01 (High range, 100 FRTN1) 7.68E-01 (High range, 200 FRTN1) 3.84E-02 (Low range, 100 FRTN1) 7.68E-02 (Low range, 200 FRTN1)
	0.04	3.69E+04 $\omega = 100$ 3.69E+05 $\omega = 10$ 1.92E+07 $\omega < 10$	7.67E-01 (High range, 100 FRTN1) 1.53E+00 (High range, 200 FRTN1) 7.67E-02 (Low range, 100 FRTN1) 1.53E-01 (Low range, 200 FRTN1)
50	0.02	2.31E+03 $\omega = 100$ 2.31E+04 $\omega = 10$ 1.20E+06 $\omega < 10$	4.79E-02 (High range, 100 FRTN1) 9.58E-02 (High range, 200 FRTN1) 4.79E-03 (Low range, 100 FRTN1) 9.58E-03 (Low range, 200 FRTN1)
	0.04	4.61E+03 $\omega = 100$ 4.61E+04 $\omega = 10$ 2.40E+06 $\omega < 10$	9.59E-02 (High range, 100 FRTN1) 1.92E-01 (High range, 200 FRTN1) 9.59E-03 (Low range, 100 FRTN1) 1.92E-02 (Low range, 200 FRTN1)

Appendix Table A1-8. Complex Modulus Limits for Cone and Plate: FRT Transducer.

PLATE DIAMETER (mm)	CONE ANGLE (rad)	G* MAXIMUM (dynes/cm <sup>2</sup> ) at Frequency $\omega$ (rad/sec)	G* MINIMUM (dynes/cm <sup>2</sup> )
25	0.1	9.22E+04 $\omega = 100$ 9.22E+05 $\omega = 10$ 4.79E+07 $\omega < 10$	9.59E-01 (High range, 100 FRT) 1.92E-00 (High range, 200 FRT) 9.59E-02 (Low range, 100 FRT) 1.92E-01 (Low range, 200 FRT)
	0.02	1.84E+04 $\omega = 100$ 1.84E+05 $\omega = 10$ 9.59E+06 $\omega < 10$	1.92E-01 (High range, 100 FRT) 3.84E-01 (High range, 200 FRT) 1.92E-02 (Low range, 100 FRT) 3.84E-02 (Low range, 200 FRT)
	0.04	3.69E+04 $\omega = 100$ 3.69E+05 $\omega = 10$ 1.92E+07 $\omega < 10$	3.84E-01 (High range, 100 FRT) 7.68E-01 (High range, 200 FRT) 3.84E-02 (Low range, 100 FRT) 7.68E-02 (Low range, 200 FRT)
50	0.02	2.31E+03 $\omega = 100$ 2.31E+04 $\omega = 10$ 1.20E+06 $\omega < 10$	2.40E-02 (High range, 100 FRT) 4.80E-02 (High range, 200 FRT) 2.40E-03 (Low range, 100 FRT) 4.80E-03 (Low range, 200 FRT)
	0.04	4.61E+03 $\omega = 100$ 4.61E+04 $\omega = 10$ 2.40E+06 $\omega < 10$	4.79E-02 (High range, 100 FRT) 9.58E-02 (High range, 200 FRT) 4.79E-03 (Low range, 100 FRT) 9.58E-03 (Low range, 200 FRT)

Appendix Table A1-9. Complex Modulus Limits for  
Couette (geometry:  $2R_C=34mm$ ,  $2R_B=32mm$ ,  $L=34mm$ ).

TRANSDUCER TYPE	G* MAXIMUM (dynes/cm <sup>2</sup> ) at Frequency $\omega$ (rad/sec)	G* MINIMUM (dynes/cm <sup>2</sup> )
100 FRTN1	3.94E+03 $\omega = 100$	8.19E-02 (High range)
	3.94E+04 $\omega = 10$	8.19E-01 (Low range)
	2.05E+06 $\omega < 10$	
100 FRT	3.94E+03 $\omega = 100$	4.10E-02 (High range)
	3.94E+04 $\omega = 10$	4.10E-03 (Low range)
	2.05E+06 $\omega < 10$	

Appendix Table A1-10. Complex Modulus Limits for  
Double Wall Couette (Geometry: Cup OD = 34 mm,  
Cup ID = 27.95 mm, Bob OD = 32 mm, Bob ID = 29.5 mm).

TRANSDUCER TYPE	G* MAXIMUM (dynes/cm <sup>2</sup> ) at Frequency $\omega$ (rad/sec)	G* MINIMUM (dynes/cm <sup>2</sup> )
100 FRTN1	5.35E+02 $\omega = 100$	1.11E-02 (High range)
	5.35E+03 $\omega = 10$	1.11E-03 (Low range)
	2.78E+05 $\omega < 10$	
100 FRT	5.35E+02 $\omega = 100$	5.57E-03 (High range)
	5.35E+03 $\omega = 10$	5.57E-04 (Low range)
	2.78E+05 $\omega < 10$	





# *Appendix B*

## **Inertia and Compliance Fixtures**

This appendix provides the inertia and compliance fixture numbers.

Appendix Table B-1. Inertia Numbers

Valid for any ARES (using V8.xx.xx firmware) with a 1KFRT or 2KFRT.

Tool	Material	Inertia (g-cm <sup>2</sup> )
8mm Cone or Plate	Titanium	25.0
8mm Cone or Plate	Steel	36.0
25mm Cone or Plate	Titanium	25.0
25mm Cone or Plate	Steel	44.0
25mm Cone or Plate	Plastic	60.0
50mm Cone or Plate	Titanium	62.5
50mm Cone or Plate	Steel	119.0
50mm Cone or Plate	Plastic	84.0
Disposable 8mm Plate	Aluminum	58.0
Disposable 25mm Plate	Aluminum	58.0
Disposable 40mm Plate	Aluminum	75.0
Disposable 50mm Plate	Aluminum	86.5
Disposable 25mm Plate	Steel	62.5
Disposable 50mm Plate	Steel	145.0
Torsion/Rectangular (old)	Steel	48.0
Torsion/Rectangular (new)	Steel	122.5
16.5mm Bob	Titanium	24.5
25mm Bob	Titanium	56.0
32mm Bob	Titanium	108.0
Double Wall Bob	Titanium	75.5

**Appendix Table B-2. Compliance Numbers**

Valid for any ARES. Requires V7.xx.xx Orchestrator software. Spring Transducer must have compliance calibration done first before using these numbers.

<b>Forced Convection Oven</b>	<b>Numbers to Enter</b>
All 316 Stainless Steel plates, cones, and disposable fixtures	0.5240 $\mu\text{R/g-cm}$
All Titanium plates, cones, and disposable fixtures	0.9300 $\mu\text{R/g-cm}$
All Invar plates and cones	0.7000 $\mu\text{R/g-cm}$
New Torsion/Rectangular tool	0.1452 $\mu\text{R/g-cm}$
Old Torsion/Rectangular tool without lower diaphragm	0.4000 $\mu\text{R/g-cm}$
Old Torsion/Rectangular tool with lower diaphragm	0.4290 $\mu\text{R/g-cm}$
Mixed 316 stainless steel plate with Titanium plate or cone	0.7270 $\mu\text{R/g-cm}$
Mixed Invar plate with Titanium plate or cone	0.8150 $\mu\text{R/g-cm}$
<b>Bath or Peltier</b>	<b>Numbers to Enter</b>
All 316 Stainless Steel upper plates or cones	0.2620 $\mu\text{R/g-cm}$
All Titanium upper plates or cones	0.4650 $\mu\text{R/g-cm}$
All Plastic upper plates or cones	0.0000 $\mu\text{R/g-cm}$
All upper Bobs	0.0000 $\mu\text{R/g-cm}$