ARES-2 Rheometer Manual: Apparatus, Startup, Shutdown, and LN2

-McCoy Group, NMT--Sept 2021-

Introduction

The ARES rheometer measures the materials response under certain stresses and/or temperatures. There are 2 ARES Rheometers: an older, tan one in Jones 130C referred to as ARES-1, and a newer, blue one in Jones 128 referred to as ARES-2. Both ARES instruments have parallel plate and torsion testing functions; ARES-2 is the easiest to add a LN2 tank to for sub-ambient experiments. This manual regards ARES-2 specifically, though there is abundant procedural overlap with the operation of ARES-1.

<u>Safety</u>

- Instrument MUST NOT be used without the compressed house-air being turned on.
- This process may involve Nitrogen in gas or liquid form. Handle Nitrogen tanks and lines with caution, and report any visible damage to these containers.
- This process involves bringing samples up to very high or very low temperatures. Avoid touching the main machine or the LN2 compressor while the test is running to prevent injury. After testing, only handle samples once they have returned to a near-ambient temperature.
- This machine has moving parts, and lots of pinch-points. Avoid touching the motor while turning it on, and take care when closing and moving the oven chamber.
- Wear gloves while handling uncured polymers. Skin sensitivities can develop upon consistent exposure.

<u>Apparatus</u>

Below is a picture of the ARES-2 apparatus. This consists of the main machine, the LN2 Dewar, and the oven. The ARES-2 software version is V6.5.8. and the Firmware Version is ARES V8.04X5.00.



Main Machine

The main machine consists of the lower motor and the upper transducer. This is where samples are clamped and tested. The motor powers any movement of the pedestal (shown to the left below) or fixtures. The air-bearing transducer senses torque in g/cm and normal force in g. Fixtures (shown to the right below) include rectangular torsion clamps and parallel plates.



Some information regarding the gap, temperature, and force can be seen on the blue screen on the main machine (pictured to the right below). However, unlike with ARES-1, there is no

capability for instrument control with this screen. Instead, the primary mechanism to control the instrument is the RSI software, the icon of which is shown to the right below.



The height of the upper fixture can be adjusted through the RSI software, as demonstrated in the "Procedure" section, or through the buttons located on the right side of the main machine (pictured below). Pressing the middle button at the same time as the up/down button increases the motor speed.



Oven

The oven is on a sliding track, and can be put to the left of the machine if ambient conditions are desirable for testing. If used during testing, it is slid right and closes over the sample-bearing fixtures. The oven has two windows; during testing, a flashlight can be pointed through one to illuminate the sample.

Within the oven, there are two RTD temperature sensors (boxed in red below). They sense the conditions of the lower and upper oven region.



The specific readings of each RTD thermocouple can be found by going to the **Utilities tab**, selecting **Service**, and then **Check Instrument Address**. The address of the lower thermocouple is 0406; the address of the upper is 040A. The acceptable difference between the two is no more than 2C.

Liquid Nitrogen LN2 Cooling

The LN2 Nitrogen system is a Forced Convection Oven (FCO), forcing gas across heating elements. There are two settings: normal cooling, where liquid N_2 is boiled by a heater that pulses every half second, and RapidCool, where the heater is on constantly. The set temperature must be lower than -125C for RapidCool to be implemented.

Instrument Handling

The ARES is a delicate instrument, and care must be taken to handle it properly. Please become familiar with the guidelines stated below.

Transducer and Motor

- The pressure to the transducer should be between 35 and 40 psi. If air is off, do not touch the transducer.
- Do not lock or unlock transducer every day. Only do this when the instrument is being moved.
- Make sure the table is not jostled while a test is running. The transducer cannot discriminate between ambient forces and sample-generated forces.
- Make sure the motor is in a mode appropriate for the specific test being performed: dynamic, transient, or steady.
- If the motor is in dynamic mode, please don't manually overpower it away from the 12 o'clock position.
- Placing an aluminum-foil "skirt" under the oven (as pictured below) can prevent liquid polymer from dripping onto the pedestal below.



Oven

- The upper fixtures will not move if the oven has not been pushed all the way to the left or right.
- If liquid polymer spills onto the thermocouples within the oven, do not attempt to clean it up. Consult Dr. McCoy, and whatever you do, **do not touch or clean the white transducer wicks.**
- Move and open/close the oven chamber gently, so as to not chip the ceramic insulation. Particularly make sure that the fixtures do not damage the ceramic.
- Do not use silver door handle to move the oven (boxed in green)- use only the black handle (boxed in red).



• Make sure the oven temperature reading is suitable for the specific test being performed. Go to Utilities in the RSI software, hover over the Service section, and select Instrument Configuration.

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Instrument Online	
Instrument Offline	
Service >	Transducer
Remote Monitoring >	Instrument Configuration
List Instrument Warnings	Print Configuration File
Set User Temperature Limit	PID Setup
Set Tool Expansion Coefficient	Mask Test Options
Set Material Density	Check Instrument Address
Change Sample Geometry Information	Upload Instrument Firmware
	Set Instrument Byte
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istrument Setup	··· Temperature Control	
emperature Control	···· Oven (Air, Chiller or LN2 Dewar.)	I
faximum Temp	[600.0 [°C]	T
Minimum Temp	·····[-150.0 [°C]	
Cooling Temp Controller Attached	🗘 None 🔹 LN2 🥂 Gas Chiller	
Auto Gas/LN2(chiller) Switching Avail.	⊂ No ເ⊂ Yes	
Temperature Loop Control	Mode 3 (RAA-Oven Air PRT, ARES-See Manual) Oven Air Temperature Sample/Taol Temp	-
Temp Calibration Table	Mode 3 (RA4-Oven Air PRT, ARES-See Manual) Mode 4 (RA4-Tool PRT)	

Temperature Control Loop is where the oven mode is adjusted. Mode 3 ("RAA Oven Air") is used during torsion test. Mode 2 ("Sample Tool") is selected if the external temperature sensor is being used. **Keep Temp Calibration Table** as **Default**.

Software

- Do not try to launch RSI twice; only one window can be active at a time.
- One RSI file must remain open for a connection to be maintained. This file will open upon launching the software, and should not be closed.
- Do not double-click on an ".rsd" files, as this will open a second RSI window.

LN2 Nitrogen Cooling:

- Please keep all doors in the room open while the LN2 is being used. This will prevent the over-dilution of oxygen in the air.
- Please keep the cooling unit behind the centerline of the main machine's pedestal.
- The LN2 tank MUST be low pressure, around 22 psi. CHECK PRESSURE OF TANK BEFORE ATTACHING.

If you have any questions or concerns, refer to an online ARES manual. Our RSI software is V6.5.8, and our firmware is ARES V8.04x5.00. Our instruments are serviced by Patel Scientific at 14 Worlds Fair Drive, Suite # K, Somerset, NJ 08873-1300. Questions that cannot be answered "in-house" may be addressed to them at jay@patelsci.com or purav@patelsci.com.

Startup Procedure

Powering On:

1. Make sure emergency-stop air valve is open (boxed in red). The pressure gage boxed in green will be used to determine air flow to the instrument.



2. Switch on the Hankison. The switch is circled in red or use the inline switch in the bookcase below.



3. Slowly turn on the main air. The action should take about 10-20 seconds. Confirm at the pressure gage near the instrument that air flow is 60-80 psi.



4. Switch on the ARES. The switch is boxed in red and is in the left rear of the main instrument. The read out on the instrument should read the temperature of the room once ready to use.



5. Launch the RSI Software. (If in doubt, open the electric box on the main machine and check the diagnostic lights boxed in red below. The second light from the top will be lit if the connection succeeds.)



6. You may now proceed with torsion or parallel-plate testing. Refer to the appropriate manual for your purposes. If the LN2 is to be used, refer to the last section of this manual.

Shutdown Procedure:

- 1. Before shutting down the machine, make sure you have saved your test data.
- 2. Turn the motor off by going to **Control** and then **Instrument Control Panel**.
- 3. Loosen the tested sample with an Allen wrench, and store appropriately.
- 4. Turn the power off using the switch in the left rear of the main box.
- 5. Slowly turn off the main air and then the Hankison using the switch in the bookcase.
- 6. Export your test data if necessary:
 - a. In the RSI software, open the test file that you would like to export. Add all the desired data to the plot. Do this by double-clicking on the plot, going to the Layout tab, and double clicking the parameters you would like to export.
 - b. It is recommended to always export Torque, Strain, deltaL CMD Strain, preTension, G', G", G*, and tan_delta. Keep the x-axis as the test default.



c. Once the plot with all the data is open, click **File** and then **Export**. Set the data to save as '.txt'. Set the desired destination of the exported file. The data is now ready to by analyzed on Excel or Kaleidagraph.

Procedure for LN2 Nitrogen Cooling

- 1. Connect the LN2 tank by tightening the metal hose from the LN2 Dewar to the tank valve labeled "Liquid".
- 2. Turn on the valve on the liquid Nitrogen tank. Make sure it is the valve connected to the metal LN2 hose.



 Go to Utilities in the RSI software, hover over the Service section, and select Instrument Configuration. Under "Cooling Temp Controller Attached", select LN2. Under "Auto Gas/LN2 Switching Avail", select Yes.

Instrument Testing Lim	ts	
nstrument Setup		
Temperature Control	··· Oven (Air, Chiller or LN2 Dewar.)	
Maximum Temp		
Minimum Temp.	[0.0 [°C]	
Cooling Temp Controller Attached	None CLN2 C Gas Chiller	
Auto Gas/LN2(chiller) Switching Avail.	• No C Yes	
Temperature Loop Control	Mode 3 (RAA-Oven Air PRT, ARES-See Manual)	-

 Under the Control menu, open Instrument Control Panel and set the environmental controller and the Liquid Nitrogen Dewar to On. Select LN2 for "Oven Pressure Source". Keep the set temperature near-ambient for now.

Environmental Control Se	ettings			
	[°C] Ma	ax=600.0°C Min=	-150.0°C	
	Oven (Air, Chiller or I	N2 Dewar.)		-
Temperature Controller	C Off @ On			
Environmentar Optimier	. C Off @ On			
Liquid Nillogen 2 -	Gas 🛎 LN2			
DAGULI ICODO			- ha	
	- I - Cattir	DOLS		1
	Motor Control Sett	ff 📀 On		
	Motor Power + -			

5. The LN2 status is shown on the RSI software, to the bottom right. Do not initiate testing until several minutes after the indicator turns from "Fill" to "Ready" in green, and not yellow. This gives the cooler time to build a surplus level of liquid Nitrogen. Keep the oven door open while the LN2 Dewar fills. This may take anywhere from 5 to 15 minutes.



- 6. Once the LN2 indicator has been "Ready" (in green) for a while, you are free to set a subambient temperature and close the oven door at will.
 - a. You may want to take advantage of the quicker RapidCool mode by going to the Instrument Control panel, setting the temperature below -125C, and turning **RapidCool** on.
- 7. You may now proceed with subambient torsion or parallel-plate testing. Refer to the appropriate manual for your purposes.
- 8. Once testing is done, close the valve on the liquid Nitrogen tank, wait 10 minutes, and then turn off the Environmental Controller and the Liquid Nitrogen Dewar in the Instrument Control Panel. Then you may shut down the instrument