Information Package to Aid Setting Up a New ROBO Apparatus

Background

Although the information provided in the ROBO Test Method DXXXX is sufficient to allow a new user to build a ROBO apparatus, this package provides (non-mandatory) information, supplementary to that in Section 6, on components and techniques that have been found suitable when setting up an apparatus for the first time. Some of the general laboratory issues associated with handling nitrogen dioxide are also discussed as are emergency shut down procedures.

The information package was provided to the laboratories that built the ROBO apparatus used in the precision round robin. Section 6 of Test Method DXXXX, however, incorporates improvements and clarifications of the method and takes precedent over the Information Package in the event there are discrepancies.

NOTE: Information dealing with hazards does not purport to address all of the safety concerns involved. It is the responsibility of the user of this information to establish appropriate safety and health practices, to determine the applicability of regulatory limitations prior to us, and to carry out hazard analysis reviews where appropriate.

Agitation System

This information[1] is supplementary to that in Section 6.6 of the ROBO Test method DXXXX; in the event of any discrepancy, the latter takes precedence.

1. General

Three components make up the agitation system:

- the fan blade (45° pitch blade turbine)
- the fan shaft (rod)
- the mixer.

Position the reactor, when initially installed in the hood, high enough from the hood bottom so that the fan shaft can be installed. The fan shaft slides up through the packing gland, squish cap and bearing; the bearing centers the shaft through the main body. Loosen the squish cap for initial installation.

The fan blade (turbine) is attached to the fan shaft by a set screw and is positioned 6 mm from the bottom of the reaction flask. To this end, first position and secure the fan shaft high into the mixer. Then install and secure the reaction flask with the proper gaskets, loosen the shaft from the mixer and slide down (carefully) to the bottom of the reaction flask. Place a mark on the shaft at the bearing, raise the shaft 6 mm and again mark the shaft from the bearing. Finally, secure the shaft back onto the mixer at the mark.

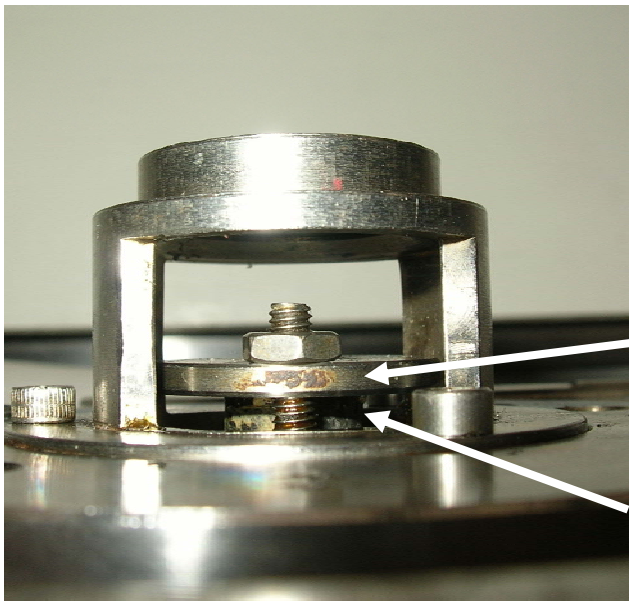
The 6 mm distance between the fan blade and the reactor bottom is optimum for the test fluid volume. This blade configuration produces a vortex for excellent temperature control throughout the entire reaction, disperses the nitrogen dioxide and provides good heat transfer from the sides of the reactor into the test fluid.

2. Installation of the packing gland

The depth of the packing gland allows several rings of packing material to be used thereby providing a good seal around the fan shaft. Use the following procedure to pack the gland:

- with the fan shaft installed, wrap one ring of the packing material around the shaft and butt the ends together; do not overlap
- offset the butted ends of the second ring of packing material 180° from the first ring
- offset the butted ends of the third ring of packing material 90° from the second ring of packing material
- lightly torque down the squish cap to produce a seal when the agitator is rotating.

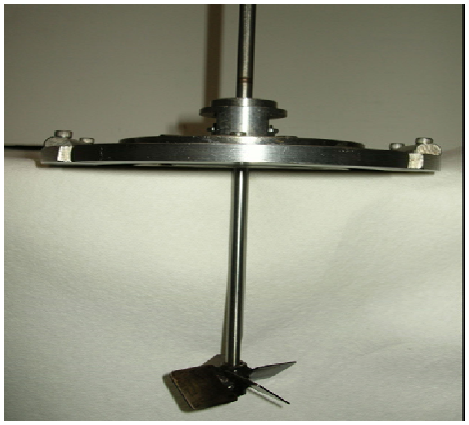
[1] Information provided by RohMax.



The squish cap, a component of the packing gland, is loosened to install the fan shaft then retightened with the agitator rotating to ensure a good seal.

Squish cap

Packing gland,



Fan shaft with blade

Heidolph[2] 2051 Brinkman mixer

[2] Heidolph is a registered trade mark of Heidolph Instruments GmbH & Co. KG, Walpersdorfer Str. 12, 91126 Schwabach, Germany. Tel: 09122-9920-68

Agitation System Components: Items Found To Be Satisfactory

Item Description	Vendor	Part number
Heidolph[2] 2051 digital mixer		2101
Fan shaft	Reimel Machine, Inc.[A]	RMI 1003 DH
Fan blade	Reimel Machine, Inc.[A]	RMI 1001 DH

[A] Available from Reimel Machine, Inc., 2575 Wyandotte Rd., Willow Grove, PA 19090.

Air Supply System

This information[1] is supplementary to that in Section 6.7 of the ROBO Test method DXXXX; in the event of any discrepancy, the latter takes precedence.

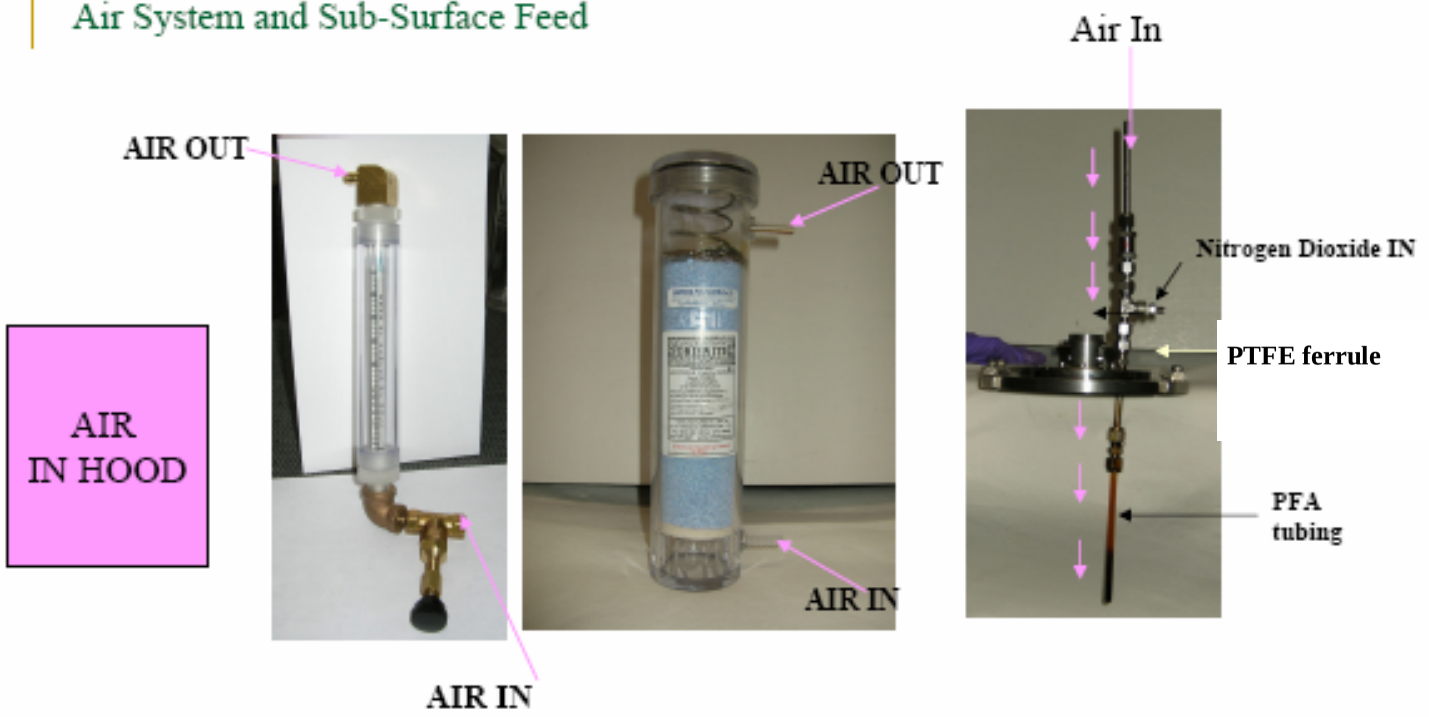
General

The air used in the reaction comes from an in-house, dried air system at 860 kPa. The pressure is then reduced by a regulator to 140 kPa and from that point 6 mm (¼ in.) braided PVC tubing is used. The flow path after the regulator is to a needle valve, a direct-read flow meter and a desiccant drier. Before entering the reactor, the nitrogen dioxide feed line is teed into the air line and the combined gases are fed subsurface in the reactor.

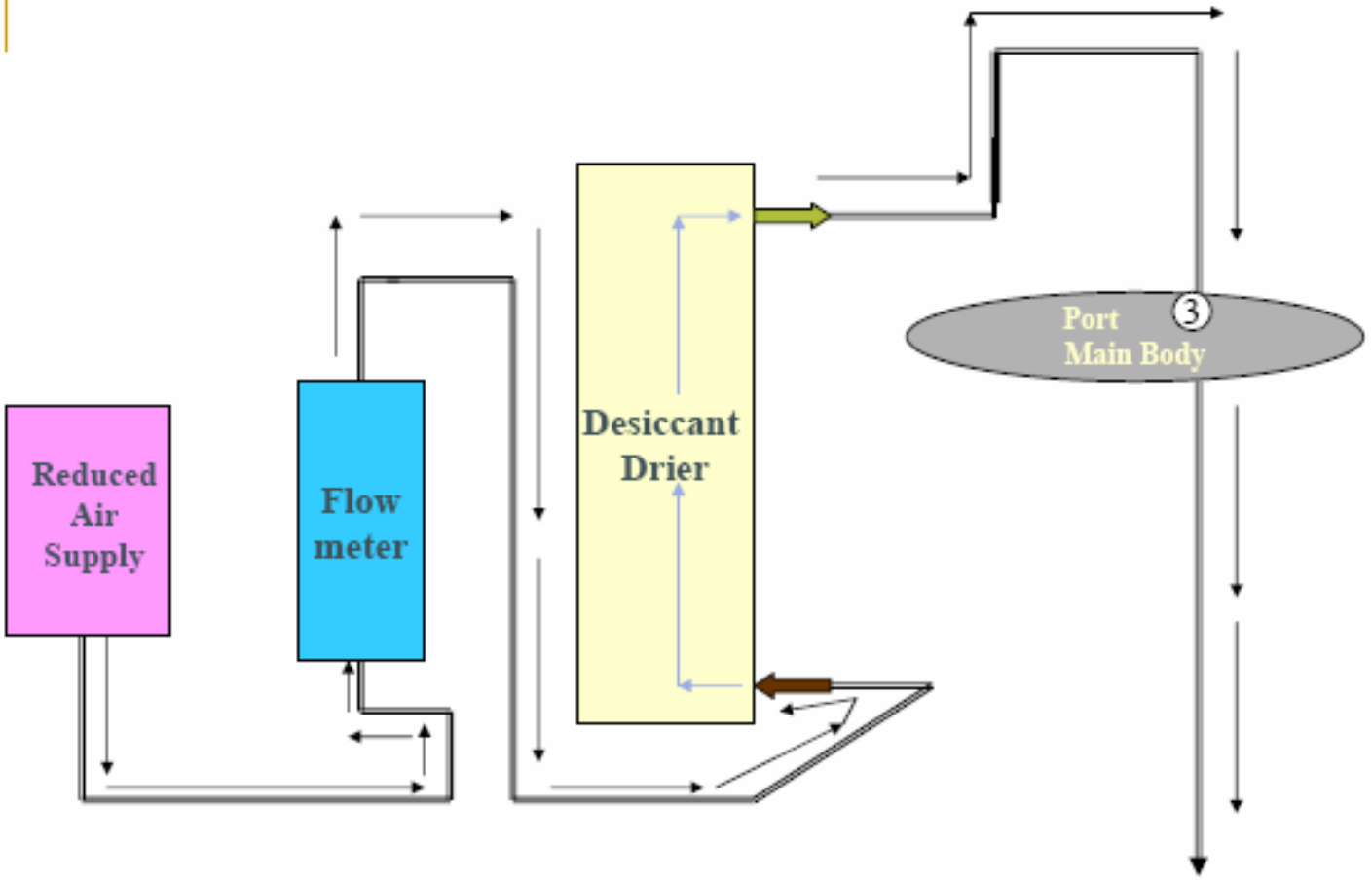
The upper gas feed tube in the reactor is stainless steel and connects to high-temperature perfluoroalkoxy (PFA) tubing which extends into the fluid to the bottom of the reactor. PFA tubing is used to prevent blockage. Prior to opening or shutting off the vacuum system, fully open the air flow to prevent oil from entering the tube to the desiccant drier. Once the vacuum pressure is set, regulate the air to test conditions for a reaction or to reduced flow when the reactor is vented.

[1] Information provided by RohMax.

Air System and Sub-Surface Feed



The air system is at a reduced pressure of 140 kPa. The air moves through a flow meter and then into the desiccant for the absorbance of any remaining moisture. Dry air is fed into the test oil using a high-temperature air tube with an outer diameter (OD) of ¼ in. Nitrogen dioxide is carried into the reactor through a high-temperature air tube. Use a PTFE ferrule for later removal.


Reduced
Air
Supply
Flow
meter
Desiccant
Drier
Port
Main Body
3

Air Supply System Components: Items Found To Be Satisfactory

Item Description	Vendor	Part number
Pressure reducer (for house air supply)	McMaster-Carr[A] Supply Company[B]	9892K21
Precision metering needle valve, 20-turn straight valve 1⅜ in. long, Type 303 stainless steel body, seat, bonnet, and needle. PTFE packing. Connections NPT female pipe size ⅛ in. Cv = 0.019	McMaster-Carr[A] Supply Company[B]	7836K25
4 in. direct-reading flow meter, 1 to 280 mL/min air, borosilicate glass tube, PTFE body and polycarbonate tube shield. Connections NPT male pipe size ¼ in. Accuracy ± 5% of full scale.	McMaster-Carr[A] Supply Company[B]	41695K22
End fittings for flow meter: **Inlet:** Brass threaded pipe fitting 90º reducing elbow ¼ in. x ⅛ in. and a nipple, threaded both ends. Pipe size ⅛ in., 1½ in. long. **Outlet:** Brass dual-barbed fitting, 90º elbow, tube ID ¼ in. Connections NPT female pipe size ¼ in.	McMaster-Carr[A] Supply Company[B]	4429K131 50785K11 44555K154
Drierite[C] gas drying jar, ¼ in. serrated nipples, 4 mm thick acrylic plastic, 2½ in. diameter x 11½ in. high and holding 20 oz of 10-20 mesh indicating absorbent. *Dry air must be used.*	Fisher Scientific[D]	09-204
Port connector tube OD ¼ in.	Swagelok[E,F]	SS 401 PC
Female tee connector, branched NPT thread, tube OD ¼ in., NPT female pipe size OD ⅛ in. for nitrogen dioxide connection.	Swagelok[E,F]	SS 10 MO 3TFT
Tube OD ¼ in. for subsurface feed, connects to female tee connector and male connector on reactor head, ferrules on male connector are PTFE.	Swagelok[E,F]	SS 304 T4W 035 20 *Stainless steel tubing*
Male connector NPT thread for reactor head, tube OD ¼ in. NPT male pipe size OD ⅛ in. bore through.	Swagelok[E,F]	SS 400 1-2BT
Union, tube OD ¼ in. for connecting high-temperature tubing to stainless steel tubing.	Swagelok[E,F]	SS 400 6
Nalgene[E] 870 PFA (Perfluoroalkoxy) tubing withstands temperatures >260º C, tube OD ¼ in.	Fisher Scientific[B]	14-176-258 pack of 25 ft

[A] McMaster and McMaster-Carr are registered trademarks of McMaster-Carr Supply Company.

[B] Available from McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100. Tel (404) 346-7000, (404) 629-6500.

[C] Drierite is a registered trademark of W.A. Hammond Drierite Company.

[D] Fisher Scientific, 2000 Park Lane Drive, Pittsburgh, PA 15275, Tel: 1-800-766-7000.

[E] Swagelok is a registered trademark of the Swagelok Company.

[F] For Sales Centers, see www.swagelok.com.

[E] Nalgene is a registered trademark of Nalge Nunc International (part of the Thermo Fisher Scientific).

General Precautions and Shut-down Procedures when Operating the ROBO Apparatus[1]

NOTE: Information below does not purport to address all of the safety concerns involved. It is the responsibility of the user of this information to establish appropriate safety and health practices, to determine the applicability of regulatory limitations prior to us, and to carry out hazard analysis reviews where appropriate.

1. General Precautions for Operating Equipment

1.1. Refer to the site Environmental, Health, and Safety (EHS) Guidelines for general safety and operating guidelines.

1.2 Inspect all glassware and Instatherm[2] heating elements for cracks and stresses before starting.

1.3. The bottom glass piece that holds the polytetrafluoroethylene (PTFE) valve plug can break easily; use caution when installing and removing it can fall out if unscrewed all the way. Check that the fluoroelastomer O-rings on the valve plug are in good condition, replacing if necessary. Before starting, check that bottom valve plug is closed.

1.4. Check that the power is off before installing Instatherm jacket cables (banana plugs).

1.5. Monitor the reactor carefully at startup, during process and while emptying.

1.6. During startup, ensure that the temperature control and agitation systems are functioning properly and that the agitator motor is actually turning the agitator - sometimes the connection becomes loose. Observe the temperature during initial heat up to make sure the controller is operating properly.

2. Emergency Shutdown Procedure of Equipment

Attend to personal protection first! Get help!

2.1. Pull all plugs from sockets at reactor hood. This will:

- Turn off all heat sources
- Turn off stirrer motor

2.2. Contain spills.

2.3. Close hood in front of reactor and contact the safety monitor.

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[1] Information provided by RohMax.

[2] Instatherm is a registered trade mark of Ace Glass, Inc.

3. Emergency Procedure in the Event of a Nitrogen Dioxide Release in the Laboratory

A release is evident by a reddish brown cloud of gas.

3.1. In the event of an uncontrolled release of nitrogen dioxide in the laboratory, do not enter the gas release area or evacuate the gas release area if in the laboratory and commence evacuation of the building immediately, by using the manual pull station by exits or by notification using the public address system in a safe area. If a person is disabled on the ground, disabled by the gas release, DO NOT ENTER AREA, notify the local fire department for assistance and rescue.

Nitrogen Dioxide

This information[1] is supplementary to that in Sections 6.15, 10.5.3 and 10.6.6 of the ROBO Test method DXXXX; in the event of any discrepancy, the latter takes precedence.

1. General

The ROBO bench method uses nitrogen dioxide, an oxidizing agent, as part of its process of simulating oil aging encountered in Test Method D7320, the Sequence IIIG/IIIGA engine test method.

The nitrogen dioxide used is > 99.5 % pure. It boils at 21.1 °C thus making consistent delivery problematical. Since the material is extremely corrosive in its pure form, all fittings, lines and valves are stainless steel or polytetrafluoroethylene (PTFE).

Our early experience with using the gas phase of the nitrogen dioxide led to inconsistent amounts delivered. A flow meter was not an option as residuals coated the meter. The alternative, using the liquid nitrogen dioxide, was also difficult because of the minute amount being delivered over time. To be consistent in measuring precise amounts of nitrogen dioxide, liquid phase nitrogen dioxide is used. Once the liquid is measured then the gas phase can be used for the reaction. This technique has proven itself in consistent amounts measured and delivered during the reaction process.

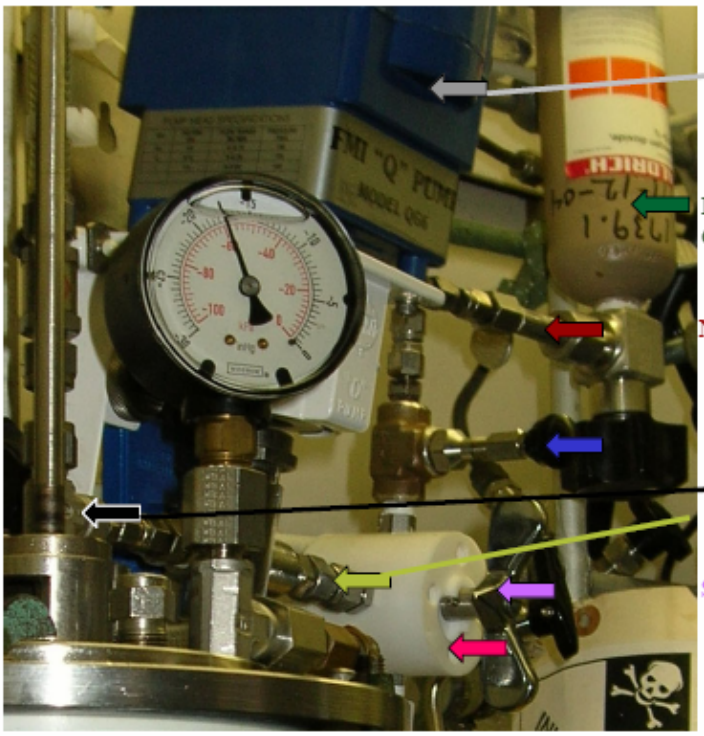
2. Nitrogen dioxide characteristics

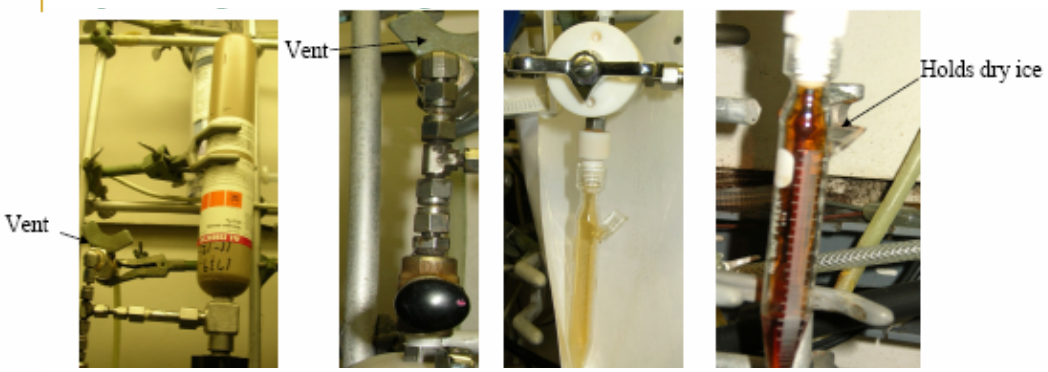
In its pure state, nitrogen dioxide is a reddish-orange-brown gas with a characteristic pungent odor. It is a corrosive and strong oxidizing agent.

Approximately 78% of air is composed of nitrogen and about 21% is oxygen. During high-temperature combustion, the nitrogen in air reacts with oxygen to produce oxides of nitrogen (NOx). Most of the NOx created during this process is nitric oxide (NO). Nitrogen dioxide (NO_2) forms when NO combines with oxygen in the air (oxidation).

CAS# 10102-44-0
Molecular weight: 92.11
Chemical formula: NO_2
Refer to the Material Safety Data Sheet (MSDS) for further information.

[1] Information provided by RohMax.

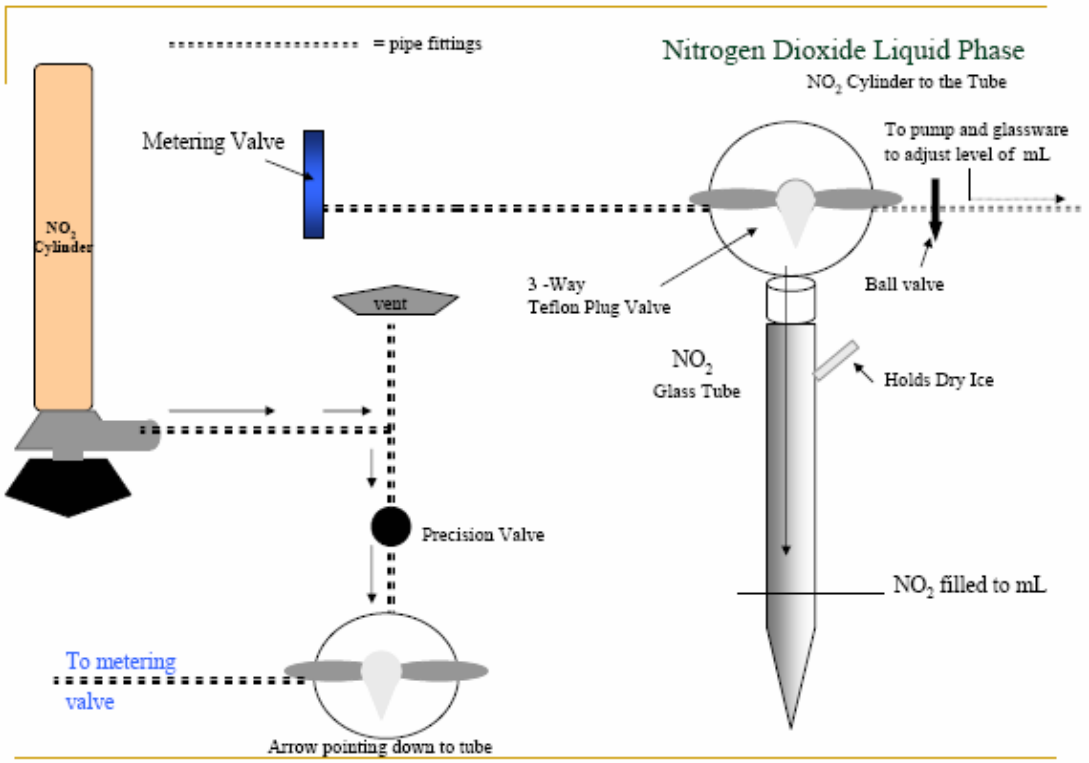

PUMP
NITROGEN DIOXIDE CYLINDER
NITROGEN DIOXIDE LINE
PRECISION VALVE
TEE CONNECTOR
NITROGEN DIOXIDE LINE
SELECTOR VALVE
3-WAY SELECTOR PLUG VALVE



Vent and inverted bottle | Precision valve | 3-way selector valve | 12-mL graduated tube

Charging the liquid nitrogen dioxide into the graduated tube (see Section 10.5.3 of ROBO Test Method DXXXX).

To extract the liquid-phase nitrogen dioxide, the lecture cylinder is installed in an inverted position. Before starting the filling process, ensure that the vent valve, precision valve and ball metering pump are closed, the (3-way) selector valve is pointing down towards the graduated tube and the high-speed metering pump is in the correct position (discharge side to round bottom).


= pipe fittings
Nitrogen Dioxide Liquid Phase
NO_2 Cylinder to the Tube
Metering Valve
To pump and glassware to adjust level of mL
NO_2 Cylinder
vent
3 -Way Teflon Plug Valve
Ball valve
NO_2 Glass Tube
Holds Dry Ice
Precision Valve
NO_2 filled to mL
To metering valve
Arrow pointing down to tube

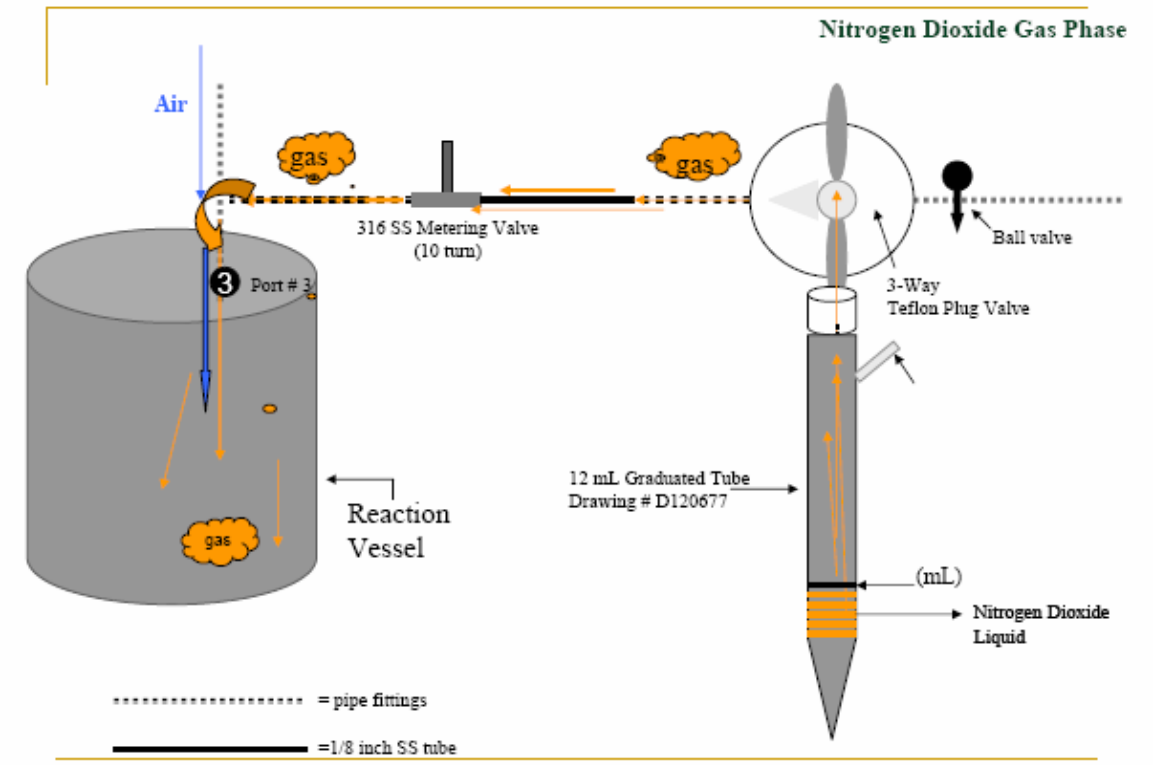
High-speed, fluid-metering pump

Glassware round bottom

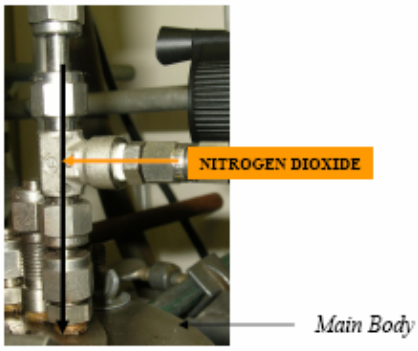
Adjusting the level of nitrogen dioxide from the graduated tube

If the desired amount of nitrogen dioxide in the tube is exceeded, the following is a safe way to pump it out:

a) Check that the high-speed, fluid-metering pump head is in the correct position (refer to pump manual) and the solution level in the round bottom is correct (the bubbler and the tube must stay in the bicarbonate solution for neutralisation of the NO_2).
b) Ensure that the selector valve points to the graduated tube.
c) Turn on the pump and open the ball valve to discharge the gas into the round bottom filled with water and bicarbonate (used for acid neutralization). The round bottom solution is changed as needed.
d) When the correct level of nitrogen dioxide is achieved:
    - Turn-off the ball valve
    - Turn-off the high-speed fluid metering pump
    - Turn selector valve towards the metering valve (see line drawing below)


Nitrogen Dioxide Gas Phase
Air
gas
gas
316 SS Metering Valve
(10 turn)
Ball valve
3
Port # 3
3-Way
Teflon Plug Valve
12 mL Graduated Tube
Drawing # D120677
Reaction
Vessel
gas
(mL)
Nitrogen Dioxide
Liquid
= pipe fittings
=1/8 inch SS tube

Air line which connects to nitrogen dioxide line which carries the nitrogen dioxide gas into the reaction flask.



Air flow into reactor

Charging the nitrogen dioxide from the tube to the reaction vessel

(see section 10.6.6 of ROBOI Test Method DXXXX)

Nitrogen Dioxide System: Items Found To Be Satisfactory

Item Description	Vendor	Part number
CGA fitting from nitrogen dioxide bottle to Swagelok[A] tube OD ⅛ in. *Fitting purchase depends on lecture bottle*		
Port connector tube OD ⅛ in.	Swagelok[A,B]	SS 201 PC
Union tee, tube OD ⅛ in.	Swagelok[A,B]	6LV 2MW 3
Port connector tube OD ⅛ in. *For vent system*	Swagelok[A,B]	SS 201 PC
Elbow 90º male connector NPT thread, tube OD ⅛", NPT male pipe size ¼ in. *For vent system*	Swagelok[A,B]	SS 200 2 4
Type 316 stainless steel ball valve, body, ball, and stem, (seats and seals are PTFE) NPT female x female connection pipe size ¼ in. *For Vent System*	McMaster-Carr[C] Supply Company[D]	45395K131
Hose fitting male connector NPT thread, tube OD ¼ in. NPT male pipe size ¼ in. *Vent Tube to Catch Bottle*.	Swagelok[A,B]	SS 400 1-2
Port connector tube OD ⅛ in.	Swagelok[A,B]	SS 201 PC
Male adapter NPT thread, tube ⅛ in., NPT male pipe size OD ⅛ in.	Swagelok[A,B]	SS 2 TA 1-2
Precision metering needle valve, 20-turn straight valve 1⅜ in. long, type 303 stainless steel body, seat, bonnet, and needle. PTFE packing. Connections NPT female pipe size ⅛ in. Cv = 0.019	McMaster-Carr[C] Supply Company[D]	7836K25
316 stainless steel threaded pipe fitting hex nipple 2 in. long, NPT male pipe size ⅛ in..	Swagelok[A,B]	SS 2 HLN 2.00
Precision selector plug valve 3-way 4-way port, solid, self-lubricating PTFE rotor plug and fluoroelastomer O-ring. Wing handles with 90º turn. Connections NPT female ⅛ in.	McMaster-Carr[C] Supply Company[D]	8163K12
316 stainless steel threaded pipe fitting hex nipple 2 in. long, NPT male pipe size ⅛ in.	Swagelok[A,B]	SS 2 HLN 2.00
12-mL graduated tube[E], adapter 5844-58 PTFE 1/8 in. NPT #7 ACE-Thred, PTFE O-ring Seal. *Request this type seal*	Ace Glass, Inc	D120677[E]
Male adapter NPT thread, tube OD ¼", NPT male pipe size ⅛ in.	Swagelok[A,B]	SS 4 TA 1 2
316 stainless steel ball valve, straight pattern, Swagelok[A] tube fitting OD ¼", Cv = 0.6 (discharge to pump for over fill and vent of graduated tube)	Swagelok[A,B]	SS-42S4
Fluid metering pump, high speed 1725 SPM, with pump head, ⅜ in. stainless steel piston (discharge to glass wear for acid neutralization)	Fluid Metering, Inc.[F]	
Male adapter NPT thread, tube OD ⅛", NPT male pipe size ⅛in.	Swagelok[A,B]	SS 2 TA 1 2
Stainless steel tube OD ⅛ in.	Swagelok[A,B]	
316 Stainless steel metering valve, straight pattern, Swagelok[A] tube fitting OD ⅛ in., 10-turn, Cv = 0.004	Swagelok[A,B]	SS SS2
Male adapter NPT thread, tube OD ⅛ in., NPT male pipe size OD ⅛ in. (connects to air supply tee)	Swagelok[A,B]	SS 2 TA 1 2

[A] Swagelok is a registered trademark of the Swagelok Company.

[B] Available from Swagelok. For Sales Centers see www.swagelok.com.

[C] McMaster and McMaster-Carr are registered trademarks of McMaster-Carr Supply Company.

[D] Available from McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100. Tel (404)-346-7000, (404)-629-6500.

[E] The sole source of supply of this component at the present time is Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ, 08362-0688 (see section 6.8 of ROBO Test method DXXXX).

[F] Available from Fluid Metering, Inc., 5 Aerial Way, Suite 500, Syosset, NY 11791. Tel (516)-922-6050.

Temperature Control System

This information[1] is supplementary to that in Section 6.9 of the ROBO Test method DXXXX; in the event of any discrepancy, the latter takes precedence.

1. General

The temperature controller maintains a constant, pre-set reaction temperature. It uses a sensor to measure continuously the reaction temperature and plugs into a timer for automatic shut-off operation. The controller output can handle resistive loads up to 10 amp (average measured amperage is 7). The temperature controller is housed in a rectangular metal enclosure, with plastic bezels on the front and on the back. All connections are made on the rear of the unit while the display and keypad are on the front. The temperature controller starts by setting the timer to the number of hours needed to run a reaction and the algorithm used is an on/off algorithm. The reaction vessel plugs into a transformer which is reduced in voltage and is read by voltmeter that plugs into the transformer output.

2. Initial set-up parameters

Thermocouple: J-type
Temperature scale: °C
Alarm set points: OFF
Audible alarm: ON
Enter advanced set-up menu: YES
Sensor offset: calibrate and change as needed
Over temperature stop above set point: 10 °C
Loop break stop: 260.0 min.
Control action: heat
ON/OFF control hysteresis: 0.1
Run time: continuous
Power up control: Stopped

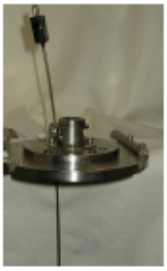
[1] Information provided by RohMax.

Temperature control system

1

2

3

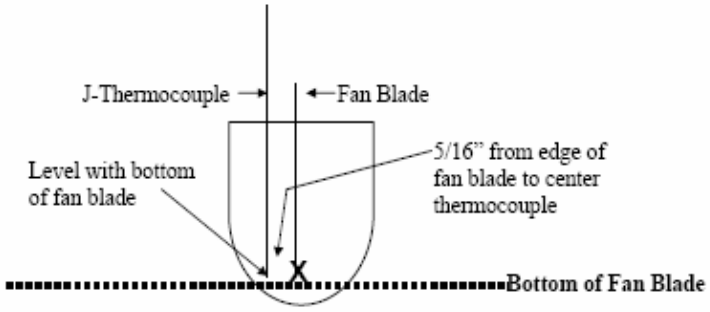


4

5

6

The temperature controller (2) plugs into the 60 h timer.
The 12 amp transformer (5) plugs into the output of the controller (2).
The reactor plugs into the transformer via a connecting cord bayonet (not shown).
The 12 in. long 1/8 in. diameter J-thermocouple (3) plugs into the transformer.
The voltmeter (6) plugs into the transformer.



J-Thermocouple placement inside reactor

Check the following settings before each run:

- the thermocouple is level with the bottom of the fan blade
- the center of the thermocouple is 5/16 in. (8 mm) from the edge of the fan blade.

As the temperature may not be uniform throughout the reactor, it is important from the point of view of precision that the temperature is always monitored and controlled at the specified position inside the reactor. When reassembling the reactor for a new run, reposition the probe, if necessary, as it is easily bent.

Temperature Control System: Items Found To Be Satisfactory

Temperature Item Description	Vendor	Part number
Timer 60 h Max resistive load 1200 W Max lamp load 600 W Max motor load 1/3 horsepower Input voltage 120VAC 50/60Hz (Temperature Controller Plugs into Timer)	McMaster-Carr[A] Supply Company[B]	1302T41
DiGi-Sense[C] Temperature Controller, 10 Ampere Output, 115VAC	Fisher Scientifi[D]	15-176-105
Variable voltage output transformer, 12 Ampere (Plugs Into Controller Output), Load rating kVA 1.44, 120VAC, Output 0-120VAC,	McMaster-Carr[A] Supply Company[B]	6994K24
3-outlet adapter (household)		
Connecting cord bayonet (from transformer to reactor)	Ace Glass, Inc[E]	9698-16
Analog panel meter, AC voltmeter, 0-50VAC	McMaster-Carr[A] Supply Company[B]	7107K513
J-Thermocouple, length 12 in., diameter ⅛ in.	McMaster-Carr[A] Supply Company[B]	39095K61
Male connector NPT thread for reactor head, tube OD ⅛ in., NPT male pipe size OD ⅛ in. bore through. (Use PTFE Ferrules.)	Swagelok[F,G]	SS-200-1-2BT

[A] McMaster and McMaster-Carr are registered trademarks of McMaster-Carr Supply Company.

[B] Available from McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100. Tel (404) 346-7000, (404) 629-6500.

[C] DiGi-Sense is a registered trademark of Cole-Parmer Instrument Co.

[D] Available from Fisher Scientific, 2000 Park Lane Drive, Pittsburgh, PA 15275, Tel: 1-800-766-7000.

[E] Available from Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ, 08362-0688.

[F] Swagelok is a registered trademark of the Swagelok Company.

[G] Available from Swagelok. For Sales Centers see www.swagelok.com.

Vacuum System Flow and Pressure

This information[1] is supplementary to that in Section 6.10 to 6.12 of the ROBO Test method DXXXX; in the event of any discrepancy, the latter takes precedence.

General

The reaction vessels are connected to an in-house (building) vacuum source with a pipe size that allows for a specific vacuum flow at the desired vacuum pressure for each reactor. The vacuum flow path starts at the reactor, passes first through a valve (to allow attainment of the desired flow rate), then to a twin chamber condenser that collects the distillate so that it does not enter the vacuum header.

To limit the pressure drop, ⅜ in. ID vacuum tubing is used to connect the components and to ensure there are no dips or sags in the vacuum line which could result in distillate collecting in the line. Inserted into the twin-chamber condenser are vacuum traps with water flow that cools and condenses vapors. The vacuum flow measurement is standard cubic feet per minute (SCFM) as described below (vacuum flow check procedure). The vacuum pressure is controlled by a relief valve and vacuum gauge for measurement reading.

[1] Information provided by RohMax.

Vacuum – Trap Components

Twin Chambered Trap

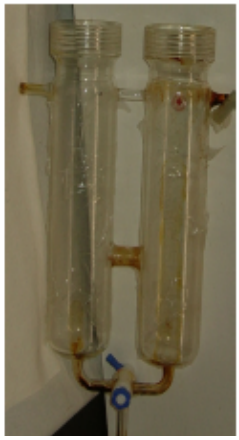


Fig. 1

Vacuum Trap

Fig. 2

Fig. 3

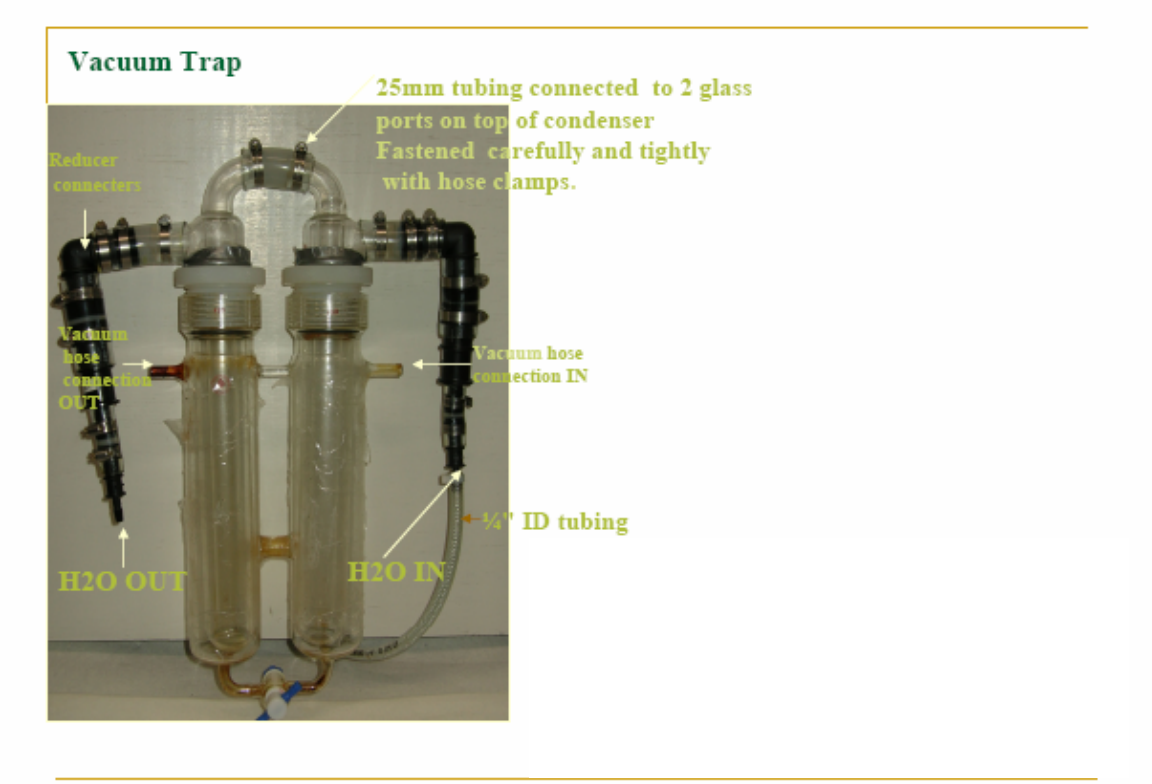
The vacuum traps (Fig. 2) fit inside the twin-chambered trap (Fig. 1), as shown in Fig. 3. The assembly of the components as seen in Fig.3 is as follows:

- Slip an O-ring (1.5 in. ID x 1/7/8 in. OD) slightly below the side tube of the vacuum trap and apply tape (e.g. duct tape). This prevents the vacuum pulling the trap further into the twin chamber and applying too much pressure on the side tube. Slip on the #50 Ace-Thred and another O-ring.
- Repeat above for the other vacuum trap.
- Connect the top ports of the vacuum traps with 25-mm tubing and carefully tighten with hose clamps. The traps are now ready to insert into the twin chamber trap and tightened with the #50 Ace-Thred.

The tubing for water is now ready for installation on the side tubes. The size of the side tube on the traps is 25mm and needs reducing to ¼ in. tubing on the outlet and inlet ports.

A water temperature is 13 °C has proved effective in condensing the vapors.

A solenoid valve plugged into a water monitor can be used to shut-down the water flow in the event of a water leak.



The twin-chamber and vacuum trap, assembled as a unit, are now ready for vacuum service.

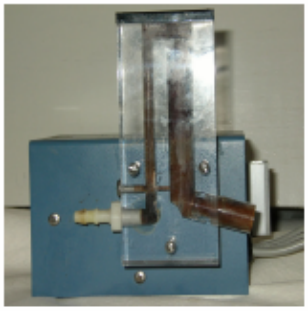
Ensure that the vacuum hose is connected to the water inlet side because this is the colder side and is where the heated vapors first enter the chamber.

Water Safety

Solenoid Valve

Water Flow Monitor

Water Flow Monitor



Vacuum Pressure Measurement

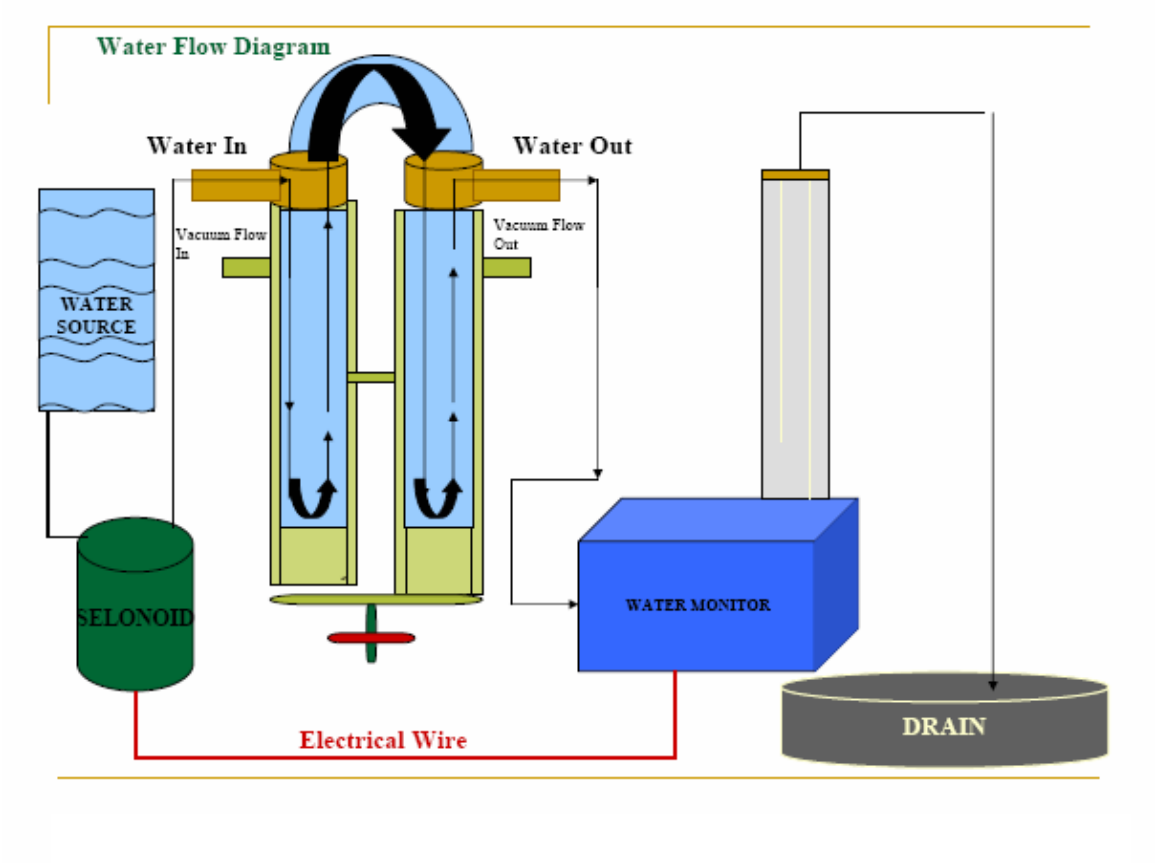
Fig. 1

Fig. 2

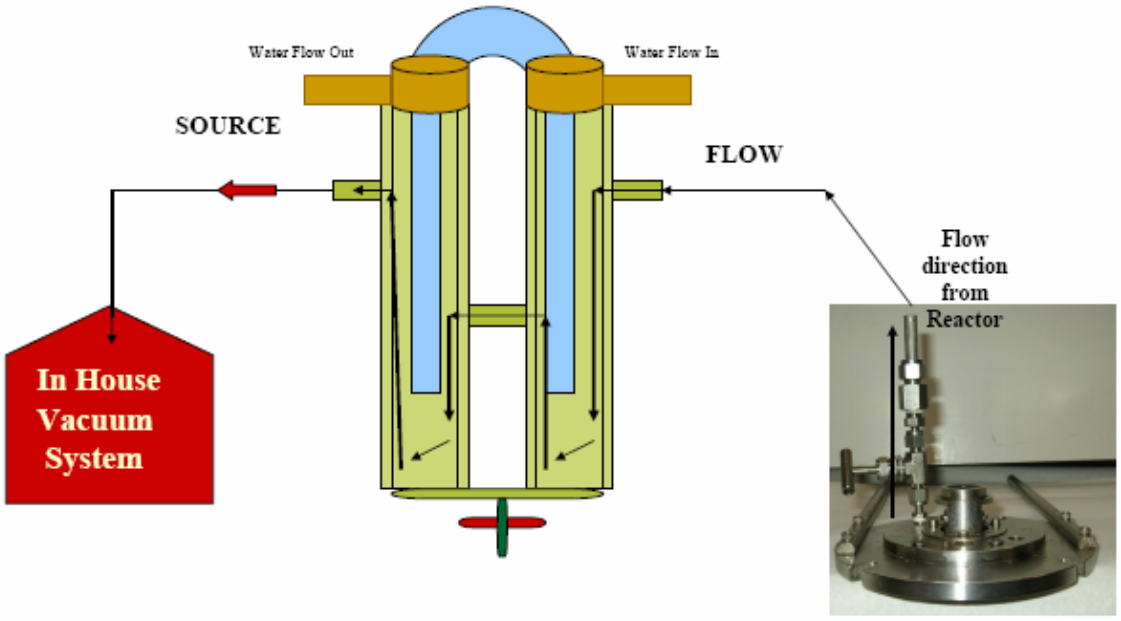
Fig. 3

The vacuum pressure can be measured using a vacuum gauge as shown above.

- Fig 1 is the relief valve
- Fig. 2 shows the gauge with the relief valve attached
- Fig. 3 shows the gauge connected and the relief valve connected to the reactor head


Water Flow Diagram
Water In
Water Out
Vacuum Flow In
Vacuum Flow Out
WATER SOURCE
SELONOID
WATER MONITOR
DRAIN
Electrical Wire

Vacuum Flow Diagram



Vacuum Flow Check

At the beginning of each test, check the vacuum flow by placing a flow meter at the top of the control valve and adjust, in combination with the vacuum relief valve, to attain the test conditions.

If the vacuum system cycles (vacuum pump shuts down), ensure the vacuum flow to the reactor is set while the vacuum pump is running.

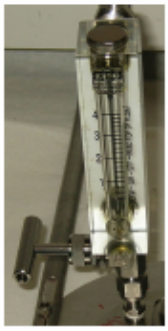
When using multiple reactors, fit each one with its own flow meter and ensure they are set together in order to obtain good repeatability between the reactors.

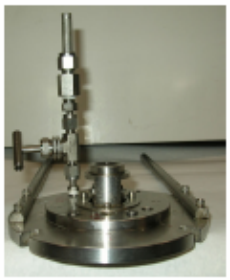
Once the valves are set and not changed they normally require no adjustment. Nevertheless, it is advisable to check them.

Check other conditions (e.g., vacuum source and reactor closure) before changing the control vacuum relief valves.

Clean the meter before each use.

Disconnect here and replace with flow meter for vacuum flow check





Vacuum System Components: Items Found To Be Satisfactory

Item Description	Vendor	Part number
Male adapter NPT thread, tube OD ¼ in., NPT male pipe size OD ⅛ in.	Swagelok[A,B]	SS4 TA 1-2
316 stainless steel needle valve with tube OD ¼ in., fitting connection, Cv = 0.37, Type 303 stainless steel body, seat, bonnet and needle. PTFE packing	McMaster-Carr[C] Supply Company[D]	45585K86
Male adapter NPT thread, tube OD ¼ in., NPT male pipe size OD ¼ in.	Swagelok[A,B]	SS 400 1-4
Female Connector NPT Thread, Tube OD ⅜ in., NPT Female Pipe Size ¼ in.	Swagelok[A,B]	SS 400 7-6
Acrylic block air flow meter 0.4 to 4 SCFM direct- reading, NPT thread female pipe size ¼ in. Accuracy ± 6 %. (Installed to adjust vacuum flow then removed)	McMaster-Carr[C] Supply Company[D]	41945K31
Male connector NPT thread, tube OD ⅜ in., NPT male pipe size ¼ in. (Installed on meter)	Swagelok[A,B]	SS 600 1 4
Elbow 90º male connector NPT thread, tube OD ¼ in., NPT male pipe size ¼ in. (Installed on Meter)	Swagelok[A,B]	SS 400 2 4
Port Connector Tube OD ¼ in.	Swagelok[A,B]	SS 401 PC
Twin chambered dry ice trap w/o fingers Two #50 Ace-Thred	Ace Glass, Inc[E]	8748-12 7506-15
45 mm diameter vacuum trap (two needed)	Ace Glass, Inc[E].	8751-20
Black fluoroelastomer O-ring (four needed) ID 1½ in., OD $1^{7}/_{8}$ in. AS568A DASH # 325	McMaster-Carr[C] Supply Company[D]	9464K47
Reducer fittings from 1 in. to ¼ in., vacuum trap water supply and return	McMaster-Carr[C] Supply Company[D]	PVC
I^2R water flow monitor		
Solenoid valve, plugs into I^2R water flow monitor	McMaster-Carr[C] Supply Company[D]	
Liquid filled 304 stainless steel case, 2½ in. dial size: grade A vacuum gauge, phosphor-bronze bourdon tube, bottom connection ¼ in. NPT male	McMaster-Carr[C] Supply Company[D]	38545K6
Adjustable brass vacuum relief valve, ¼ in. NPT male, bottom connection, vacuum rating 0 to 27 in. Hg. Type 302 stainless steel spring and type 440C stainless steel ball as a poppet	McMaster-Carr[C] Supply Company[D]	48935k25
316 stainless steel tee, ⅛ in. NPT, female x female x male.	Swagelok[A,B]	SS 2 TSW 3
⅜ in. ID black vacuum hose	Fisher Scientific[F]	

[A] Swagelok is a registered trade mark of the Swagelok Company.

[B] Available from Swagelok. For Sales Centers see www.swagelok.com.

[C] McMaster and McMaster-Carr are registered trademarks of McMaster-Carr Supply Company.

[D] Available from McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100. Tel (404) 346-7000, (404) 629-6500.

[E] Available from Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ, 08362-0688.

[F] Available from Fisher Scientific, 2000 Park Lane Drive, Pittsburgh, PA 15275, Tel: 1-800-766-7000.

Vessel Head and Reactor

This information[1] is supplementary to that in Sections 6.3 and 6.4 of the ROBO Test method DXXXX; in the event of any discrepancy, the latter takes precedence.

General

The vessel head is a stainless steel plate with several threaded ports to allow filling and for the attachment of air and nitrogen dioxide, vacuum control and relief valves and the temperature probe; it also has a center hole for the agitator shaft. Extension rods allow the plate to be clamped onto the lattice support rods inside the hood. A machined polytetrafluoroethylene (PTFE) alignment seal ring (refer to drawing) fits into a groove on the underside of the plate for centered attachment of the glass vessel; a fluoroelastomer gasket fits between the alignment ring and the glass reactor. The 1-L reaction vessel is attached to the main body by a support ring clamp and attached to the hood lattice support rods.

A stopcock valve at the bottom of the reaction flask allows the test fluid to be drained from the reaction vessel. A banana plug at the back of the reaction vessel allows an electrical cord to be plugged into the variac.

[1] Information provided by RohMax.

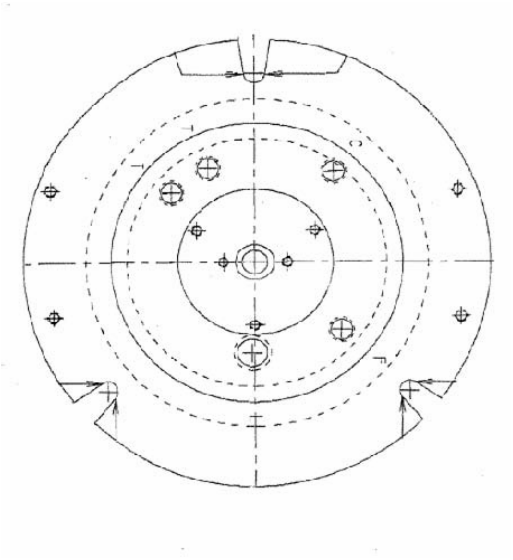
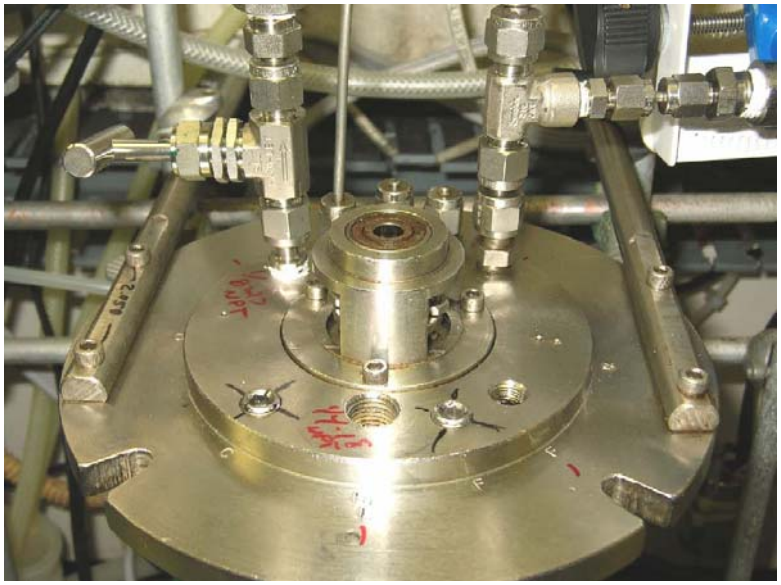


Diagram of vessel head



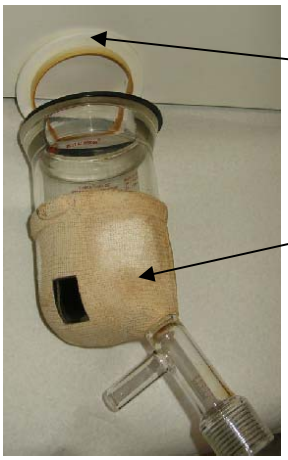
Vessel head

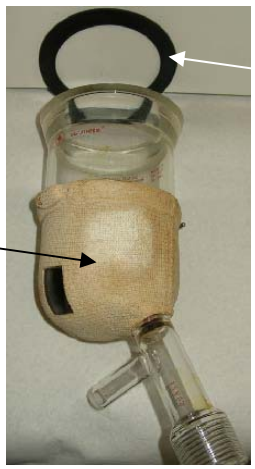
Squish cap

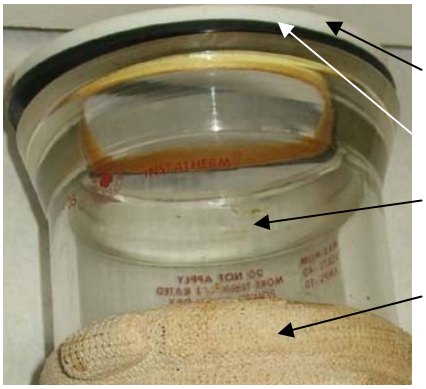
Support clamp

Stainless steel main body

Reaction vessel







[1] Instatherm is a registered trade mark of Ace Glass, Inc.

Vessel and Vessel Head Components: Items Found To Be Satisfactory

Item Description	Vendor	Part number
1-L reaction flask[A] ,bottom stopcock valve	Ace Glass, Inc.	D120676[A]
Standard clamp	Ace Glass, Inc.[A]	D124780
Black fluoroelastomer O-ring for stop cock	Ace Glass, Inc.[A].	
Machined PTFE alignment seal ring	Reimel Machine, Inc[B]	RMI 1007 DH
Fluoroelastomer gasket to fit alignment ring	Made in-house	N/A
Reaction vessel head (see drawing)	Reimel Machine, Inc[B]	RMI 1002 DH
Extension rods (see drawing)	Reimel Machine, Inc[B]	RMI 1006 DH
Cap body (see drawing)	Reimel Machine, Inc[B]	RMI 1004 DH
Squish cap (see drawing)	Reimel Machine, Inc[B]	RMI 1005 DH
Soft PTFE filament packing	McMaster-Carr[C] Supply Company[D]	9469K21

[A] The sole source of supply of this component at the present time is Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ, 08362-0688 (see section 6.3 of ROBO Test method DXXXX).

[B] Available from Reimel Machine, Inc., 2575 Wyandotte Rd., Willow Grove, PA 19090.

[C] McMaster and McMaster-Carr are registered trademarks of McMaster-Carr Supply Company.

[C] Available from McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100. Tel (404) 346-7000, (404) 629-6500.

Support clamp