Thermal *Dynamix*™, Inc.
Ammonia Dissociator

Electrically heated
Model AD10E

Thermal *Dynamix*™, Inc.
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All Thermal Dynamix electrically heated dissociators, regardless of size and capacity, are substantially identical in operation and behavior. The process of dissociating or “cracking” ammonia vapor into its constituent parts is, from an operating standpoint, relatively simple. For that reason, this manual is applicable to all TDI electrically heated dissociators.

Introduction instructions

Unless otherwise requested TDI ammonia dissociators are normally shrink wrapped for protection from the elements during transit. The integral dissociator base eliminates any need for a shipping skid or cribbing. On the larger units (6000 cfh and above) the heat exchangers and preheat chambers may be packed and shipped separately.

The TDI dissociator sits upon a full steel plate supported by its own rugged steel chassis. It is recommended and assumed the unit will be located on a non-combustible, level floor. It is also assumed the unit will be moved into its operating location before being unpackaged.

Unwrapping or Uncrating

- Carefully inspect the unit and all components for possible damage in transit. Immediately report any components that were damaged in transit to the freight carrier for reimbursement. Contact Thermal Dynamix for replacement parts.

  Note: Even though TDI may have assisted in the shipping arrangements and may have suggested a freight carrier as appropriate, the dissociator was sold FOB your truck at our dock. Ownership, title and risk of loss was transferred to you as soon as the dissociator was loaded on the freight carriers truck.

- Set aside, for future use, the separately packed components.

- The control panel enclosure contains the temperature controls, SCR power controller, main fuse disconnect, safety contactor, control relays and alarm lights and horn. These components are all hardwired in place and are ready for operation. Check them to ensure none has come loose during shipment.

- Two thermocouples and their leads are also packed in the panel enclosure.

- The silicon carbide heating elements are packed and shipped separately. **Handle these elements very carefully. They are very fragile and break easily.** Once they’re installed they will last a very long time. The big danger is handling them during installation.

Installation and Set up

There are normally five connections required in the installation of a TDI ammonia dissociator (seven connections if water cooling is used) and the silicon carbide heating elements must also be installed. These connections are:

- Inlet feedstock pipe w/shut-off (for anhydrous ammonia vapor)
- Output process gas pipe w/shut-off (for dissociated ammonia gas)

FIG. #1

- Vent pipe w/shut-off
- Relief valve vent(s). These must be piped directly to outside with no obstructions or shut-offs.
- Electrical power feed
- (Cooling water inlet and outlet are the sixth and seventh connections where required)

Generally speaking, and not at all critical, the sequence of events is as follows:

1. Connect the inlet feedstock pipe to the inlet of the dissociator gas train just before the inlet ball valve. Use schedule 80 steel for this connection. Use special joint compound suitable for ammonia and steel fittings.
2. Connect the process gas output pipe, just after the output ball valve, to the pipe leading to the furnace being served by the dissociator. Use special joint compound suitable for ammonia and forged steel fittings.
3. Connect the vent pipe, just after the vent ball valve shut-off, to a pipe connected to the outside of the building. Use special joint compound suitable for ammonia and steel fittings. The relief valve(s) may be connected to this vent line provided the connection(s) are <u>after</u> the vent ball valve shut-off. <u>The relief valve(s) must be piped directly to the outside with no valves or obstructions in the line.</u>
4. If water cooled heat exchangers are installed connect the inlet and outlet of the cooling water pipes just before and after the ball valves in the cooling pipes.
5. Remove the turret top cover and carefully place the six ceramic heating element sleeves into the element ports in the turret of the heating chamber and **very, very** carefully insert the silicon carbide heating elements into their insulating sleeves. (See fig. #2A)
6. Attach the power feed wires to the heating elements as shown in fig.#2B.
7. Insert the TC's into the two compression fittings on the retort chamber directly behind the panel enclosure. Their leads are then plugged into the panel jack at the bottom of the panel enclosure. Push the TC's into the chamber as far as they will go and then withdraw them about ¼". Tighten the compression fitting. The top TC is for the temperature over-limit control. The bottom for the temperature control.
8. Connect a main power feed, 480V, 3 Ph, 60 Hz, to the main fused disconnect in the panel

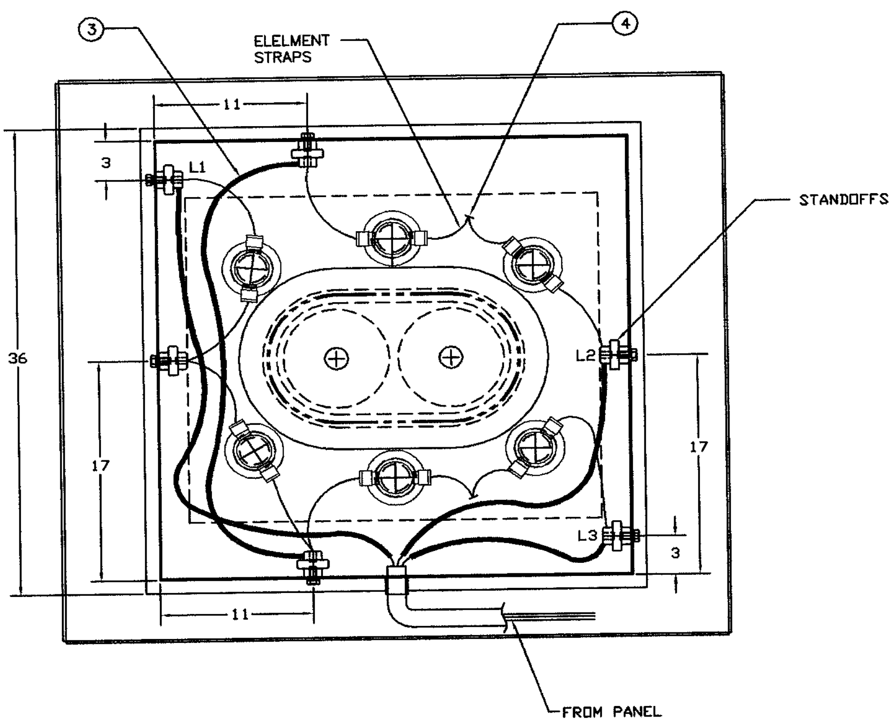
enclosure.

There are some cautionary notes here that relate to the set up and operation of the dissociator and also the operation and supply of dissociated ammonia gas to a furnace:

- The inlet pressure of the ammonia vapor supplied to the inlet ball valve of the dissociator must not exceed 35 psig. The dissociator retort maximum operating pressure is 5 psi. The system relief valve is set for 8 psi. Do not operate the system at higher pressures as the risk of retort rupture increases dramatically with an increase in pressure.
- There should be a safety relief valve on the ammonia feed line, before the dissociator inlet, to ensure the supply line ammonia pressure doesn't exceed 35 psig. That relief valve should be vented to a line leading directly to the outside of the building. That relief valve is ***not*** the responsibility of TDI. It's the responsibility of the user/buyer to ensure the pressure doesn't exceed 35 psi.
- If a single vent line is used it must be sized to handle all the relief valves connected to it.
- Make sure the process gas supply pipe to the furnace is adequately sized to avoid an excessive pressure drop between the dissociator and the furnace.
- Do not admit atmosphere (dissociated ammonia gas) to a furnace if the furnace temperature is

FIG. #2A

FIG.#2B



below 1400°F.
- Ammonia is extremely unpleasant stuff that can very quickly do a great deal of damage to human skin and tissue. The handling and use of ammonia should be by persons trained in its characteristics. At the very least, one should wear appropriate, tight fitting goggles, a face mask and rubber gloves.

Operating Instructions

The TDI Ammonia dissociator is a relatively simple machine. Its operation is straight forward and uncomplicated. There are, however, slightly different procedures for starting up a new dissociator (or one with a new or freshly charged retort) and starting a dissociator that has been in use. The need for different procedures is because new catalyst must first be “activated” in order to prepare it so it will accelerate and sustain the dissociation process.

In this manual the term “new start” refers to starting a machine with new or fresh catalyst, regardless of how old the actual dissociator is.

The term “restart” refers to starting a machine that has serviceable, previously activated catalyst but has been out of service for a brief period of time. This machine has not been disconnected or had its piping interrupted. It may be idling at some nominal “warm” temperature.

The term “cold start” refers to starting a machine that has serviceable, previously activated catalyst but has been out of service for an extended period of time and may have had some of its plumbing open for maintenance or may have been exposed to outside weather conditions.

A. New Start

There are several things to appreciate in a new start of a new dissociator:

- There is probably going to be some moisture in the chamber insulation. The initial heat-up should be done in stages to allow the insulation to “bake out”. See the Bake out schedule on page 13.
- The start-up of a new dissociator, or a dissociator with new catalyst, is going to actually make some water in the retort.
- The water produced during the activation process will irritate your skin. Be prepared to wash your hands quickly if you get any splashed on your skin. It’s a good idea to wear goggles and rubber gloves while checking and draining the water produced during the activation process.

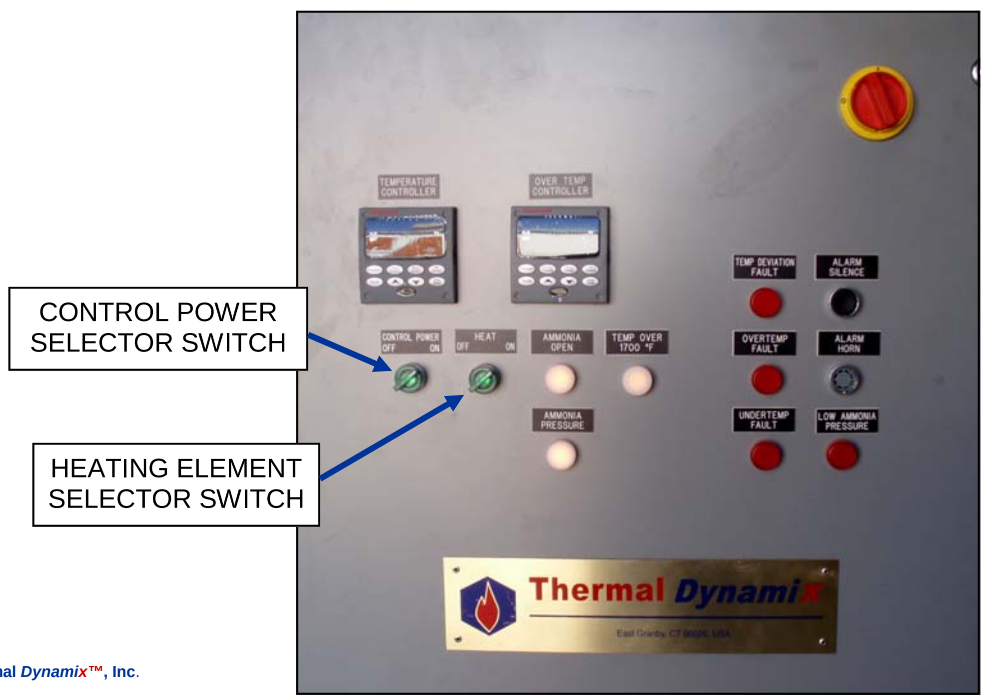


FIG. #3

- There may be a considerable amount of moisture in the dissociator heating chamber. It will come off as steam as the unit heats up. As it evaporates it may have a pleasant, sweet smell, no odor at all or an unpleasant smell. It depends upon what was used as a slurry vehicle by the insulation maker. In any event the odor will go away as soon as the insulation dries.

It is useful to understand in the starting up of a new dissociator, or one that has new catalyst in the retort, that in order to prepare that new catalyst for service it must first be "activated" (the chemical

FIG. #4

FIG. #5

term is "reduced"). This activation process may take 2 to 4 hours; this is *additional* time required after drying out the insulation.

Nickel catalyst is comprised of approximately 7% nickel oxide. In order for the active ingredient, nickel, to be available to start the ammonia dissociation process the oxide has to be gotten rid of in a process called "reducing". This happens as the hydrogen in the ammonia vapor combines with the oxygen in the oxide producing water.

1. Close the ammonia vapor inlet ball valve. (fig.#1)
2. Close the process gas outlet ball valve. (fig.#1)
3. Open the process gas vent ball valve. (fig.#1)
4. Turn the control power on (The green light will show and the alarm will sound). (See fig. #3)
5. Set the controller to 300°F (The heater contactor will be energized and the blue light will show).
6. Allow system to soak at 300°F for two hours.
7. Reset the temperature controller to 600° and allow to soak for one hour.
8. Reset the temperature controller to 1000° and allow to soak for one hour.
9. Set the temperature controller to 1750° and wait for set point to be reached.
10. Open the ammonia vapor inlet ball valve. Check the ammonia supply pressure and ensure it does not exceed 35 psig.
11. Manually open and latch the process gas safety shut-off valve open, allowing ammonia vapor to flow into the retort, through the system and discharge to the vent. (fig.#6)
12. Check the system pressure at the low pressure gage just after the system pressure regulator. Set the regulator to provide a system pressure of between 3 and 5 psig.
13. Set the flow control needle valve in the flow meter to about 10% of the dissociator capacity. (fig.#5)
14. Open and check the 1/4" drain in the drip leg immediately before the flow meter inlet. Allow any collected water to drain and then close valve. This is the time water will begin to appear in the process gas output lines because there is now hydrogen present (in the ammonia) to begin combining with the oxygen (in the catalyst oxide). This drain must be opened and checked repeatedly during this catalyst activation process. It's not unusual for the system to produce 2 to 4 quarts of water during this new startup. Once the production of water stops the activation is

complete and the drain is no longer necessary.
15. Once the production of water stops the flow control needle valve in the flow meter may be set to the desired flow.
16. Open the process gas output ball valve.
17. Close the process gas vent ball valve.

B. Restart (of an idling, warm or previously operating dissociator)

1. Close the ammonia inlet ball valve.
2. Close the process gas outlet ball valve.
3. Open the process gas vent ball valve.
4. Turn the control power on (The green light will show and the alarm will sound).
5. Set the temperature controller to 1750° or whatever previous set temp was used (The heater contactor will be energized and the blue light will show). Wait for the temperature to come up to the set point.
6. Open the ammonia vapor inlet ball valve. Manually open and latch the process gas safety shut-off valve open, allowing ammonia vapor to flow into the retort, through the system and discharge to the vent.
7. Set the flow control needle valve in the flow meter to the desired output flow.
8. Open and check the 1/4” drain in the drip leg immediately before the flow meter inlet. Allow any collected water to drain and then close valve (there should be little or no water present).
9. Open the process gas outlet valve.
10. Close the process gas vent valve.

C. Cold start

1. Close the ammonia inlet ball valve.
2. Close the process gas outlet ball valve.
3. Open the process gas vent ball valve.
4. Turn the control power on (The green light will show and the alarm will sound).
5. Set the temperature to 900° and allow to soak for one hour.
6. Raise the temperature controller to 1750° (The heater contactor will be energized and the blue light will show). Wait for the temperature to come up to the set point.
7. Open the ammonia vapor inlet ball valve. Manually open and latch the process gas safety shut-off valve open, allowing ammonia vapor to flow into the retort, through the system and discharge to the vent.
8. Set the flow control needle valve in the flow meter to the desired output flow.
9. Open and check the 1/4” drain in the drip leg immediately before the flow meter inlet. Allow any collected water to drain and then close valve (there should be little or no water present).
10. Open the process gas outlet valve.

FIG. #6

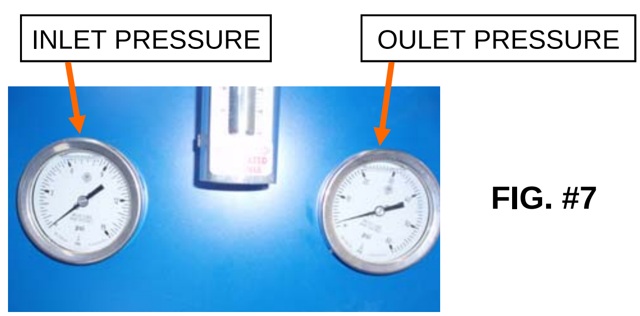


FIG. #7

11. Close the process gas vent valve.

Shutting Down the Dissociator

1. Manually release the safety shutoff valve to the closed position. This will close the process gas to the dissociator.
2. Close the dissociator ammonia supply line valve to the furnace.
3. Close the ammonia inlet ball valve.
4. Shut the control power off.

The vent line ball valve may be left open. It is our suggestion that if the unit is to be shut down for a long period of time the vent line valve should probably be closed. This will minimize the possibility of getting moisture into the system.

Maintenance

- Regular maintenance is very important for atmosphere generating equipment. The flow scope, pressure regulator, and other components are precision instruments and any contamination will impair the proper functioning of these units. Under no circumstances should oil or other petroleum-based compounds be used as valve lubricants or anywhere else in the piping system.

- It's important that the thermocouples and temperature controls be accurate. It is recommended they be checked and calibrated at least once every year.

- For efficient operation of the dissociator and the safety and well being of those working in the vicinity of the dissociator all pipe joints must be absolutely gas tight. Periodically check all unions and joints. If an unsatisfactory dew point is being obtained the pipe joints are the first thing to check.

- The pressure relief valve is actuated only in an unusual condition and normally requires no maintenance. If, however, one should open to relieve a high pressure condition the dissociator must be shut down immediately. When the cause of the excess pressure has been identified and resolved, normal operation may be resumed.

A. Changing the Retort and/or Preheat Chamber

To remove the retort assembly from the dissociator; un-bolt the flanges connecting the pre-heat chamber to the retort piping and the heat exchanger and remove the pre-heat chamber. Un-bolt the cover and lift the cover and retort as a unit by lifting straight up by the lifting strap provided until it clears the retort chamber. Swing cover to one side. It is extremely important the cover and retort assembly be lifted straight up as a unit. The retort must not be allowed to swing as it will strike and break a heating element. After the retort is out of the chamber the cover may be removed from the retort pipes.

When installing a retort assembly first install the cover and then very carefully lower the retort into the chamber by its lift strap; taking great care to ensure it does not swing into one of the heating elements. Use of the lift strap will greatly aid in allowing the retort to remain plumb as it is lowered into the chamber. The wedge placed in the keyhole in the lift strap determines the proper location of the retorts in the chamber and supports the retorts at the cover. The split collars should be installed on the retort inlet and outlet pipes so that the collars also bear upon the cover. The split collars, in addition to providing additional support security, reduce the potential for the retort to "swing".

We cannot emphasize enough the importance of using care in lifting the retort out of, and lowering it back into, the chamber. Failure to use care almost guarantees the breakage of an element.

B. Changing a heating element

The silicon carbide heating elements may be changed individually and, with the exception of the top cover plate, without removal of any other component. **Great care must be exercised in the removal of a heating element because they are very fragile.** Each element weighs about 10 pounds and is supported at the top in a ceramic sleeve. It must be withdrawn and inserted straight up and not allowed to scrape along the sleeve. It has been our experience that the overwhelming instances of breakage of the silicon carbide elements occur in the installation or removal of the element. If you can get the element to survive the installation process it will last a long, long time.

Design Principles

An Ammonia Dissociator will produce a 75% hydrogen, 25% nitrogen atmosphere. The quality of the atmosphere depends to a large degree upon the purity of the ammonia fed into the dissociator. The degree of dissociation depends upon the temperature of the retort, the surface area of the catalyst and the rate at which the ammonia vapor is fed to the dissociator. Normally, at capacity, an operating temperature of 1750° F produces a gas containing less than 50 parts-per-million of residual ammonia. Operation at flow rates less than capacity will produce less residual ammonia. Increasing the temperature will also reduce the residual ammonia in the process gas. To that end the retort chamber may be operated at up to 1850°F. Having said that we do recommend an operating temperature no higher than that necessary to yield the desired residual ammonia levels. Adjust the temperature controller set points accordingly.

Safety

There is great hazard involved in the use of poisonous and explosive gas atmospheres. Certain precautions must be observed to prevent injury to personnel or damage to equipment.
The minimum ignition temperature for dissociated ammonia gas (in the presence of air) is approximately 11OO° F. Allow 300° F safety margin. Do not attempt to introduce dissociated ammonia atmospheres into furnaces at temperature lower than 1400° F.
Before proceeding with furnace repairs, disconnect the atmosphere supply lines to the furnace and thoroughly purge by circulating air through the furnace.

The dissociator is equipped with temperature and pressure safeguards. These should be checked periodically by creating the conditions which normally cause the safety devices to function (i.e., low temperature, over temperature, under pressure, over pressure, etc). A program of planned maintenance will ensure trouble free service and the procedures outlined in Section III should be followed.

This electrically heated ammonia dissociator is equipped with the following:

A. A low pressure relief valve is furnished immediately down stream of the ammonia dissociator pressure regulator. Should the regulator fail, the relief valve is set to open at 8 psig and vent the ammonia vapor gas to the roof.
B. An ammonia vapor gas safety shut-off valve with manual reset and electric holding coil is installed on the incoming ammonia vapor gas line (point "C"). See Flow Diagram included with this manual. A power failure or an over-temperature condition, or an under-temperature condition within the dissociator will cause the safety shut-off valve to close off the supply of ammonia vapor gas. The safety shut-off valve cannot be manually latched until the fault has been corrected.
C. An over-temperature set of contacts in the control instrument is provided. These contacts are factory set for 1850º F. An over-temperature condition will de-energize the safety contractor, sound an alarm, causing the heater to be de-energized, and close the ammonia vapor gas safety shut-off valve. A light has been provided to indicate the over-temperature control contacts are the cause for the alarm.

D. As additional protection, temperature interlocks are provided as follows:

   a. The safety shut-off valve cannot be latched to admit ammonia vapor until the dissociator temperature is up to 1700º F.

   b. At 1850° F an over-temperature fault occurs, which shuts down the heat and disables the MRV solenoid. If an over-temperature condition is experienced, the switch must be manually reset. The supply of ammonia vapor is closed through the safety shut-off valve.

E. Incorporated in the temperature control instrument is a deviation alarm, which is used to alert operators that temperature has deviated away from set-point. This alarm is indicated with audio and visual signals. The heat and/or the atmosphere are not disabled as a result of the alarm. The deviation alarm is used to alert the operator of a potential problem before the system is shut down as the result of a temperature problem.

NEW UNIT BAKE OUT SCHEDULE

The insulation assembly in a Thermal Dynamix dissociator is comprised of two elements: The heating chamber walls are of a vacuum formed material which is about 2 inches thick. This is backed up with about 8 inches of bulk ceramic fiber.

Since the actual chambers are formed from a wet slurry type material (poured into forms, much like cement) they require some time to dry and cure. They may very well have some entrained moisture when we install them. The probability of moisture is greatest in the chamber walls because of the way in which they are fabricated.

In order to ensure all the moisture is eliminated from the insulation of a new dissociator it is a good idea to heat the unit up slowly the first time it's used. We suggest the following schedule as a reasonable approach:

1. Bring the chamber temperature up to 200°F and let it soak for one hour at that temperature.
2. Increase the chamber temperature from 200˚F to 500˚F at a rate of 200˚F per hour and let it soak for two hours at that temperature.
3. Increase the chamber temperature from 500˚F to the normal operating temperature of 1800˚F at a rate of 300˚F per hour.

The first time heat up of a new dissociator, using this schedule, will require about 6 hours or so. We think it's time well spent and, since it's only needed on the initial start-up, we strongly recommend the schedule be followed.

Recommended Spare Parts List

1000 SCFH AMMONIA DISSOCIATOR

Part Number	Recommended Quantity
Heating Element (6 Required)	2
Collar, Split	2
Thermocouple Element with Insulators	2
FOIL-2HT Oil, Flow Scope	1 Ea. 6 oz bottle

Included Drawings: (located in back pocket of folder.)

DWG. #AD10E-01 GENERAL ARRANGEMENT

DWG. # AD10E-02 FLOW DIAGRAM

DWG. #AD10E-1 ELECTRICAL SCHEMATIC (1 THRU 5)

Vendor Data

Control Concepts: SCR power controller

Honeywell: Temperature control

Honeywell: Over-temp control

Hydroseal: Pressure Relief Valve(s)

Fisher: Pressure regulator