

PD-Series Nafion[®] Dryer Cleaning Procedures

Overview

Performance of PD-Series dryers may decline over time due to buildup of residues on the inside or outside of the Nafion tubing within the dryer. This procedure describes the various steps required to clean a PD-Series dryer to restore it to normal performance.

General Description

PD-Series dryers contain multiple strands of Nafion tubing with appropriate fittings at each end for connection of gas lines. A shell tubing of inert material surrounds the Nafion tubing, permitting purging of the Nafion with dry gas. Fittings for connection of a purge gas to each end of the shell are also included.

PD-Series dryers may be constructed with:

- a) Various numbers (50, 100, or 200) of strands of 0.030" OD Nafion tubing
- b) Nafion tubing of various lengths (12", 24", 48", or 72")
- c) Most headers are constructed of cast epoxy, some have molded polypropylene (PP)
- d) Shell/fittings are of various materials (PP, Teflon-coated aluminum or stainless steel SS)
- e) Various flow rate capacities up to 40 liters per minute.

Failure of dryers during operation may occur due to three causes:

- a) Collapse of the tubing due to negative sample pressure
- b) Introduction of liquid water into the tubing rather than water vapor
- c) Clogging of the tubing, internally or externally.
 - 1) Physical blockage of the tubing will prevent sample flow.
 - 2) Occlusion of the surface of the tubing (externally with oil from the purge air or internally with deposits from the sample) will prevent water from permeating through the tubing wall.

Cleaning of a PD-Series dryer requires the following general steps:

- a) Remove the dryer element (Nafion tubing bundle) from the housing.
- b) Remove any visible deposits from the ends of the headers.
- c) Rinse the dryer element in an appropriate solvent
- d) Dry the dryer element by blowing dry air through it.

Or

- c) Soak the dryer element in 10% nitric acid at 80°C for 2 hours.
- d) Rinse the dryer element with DI water to remove the acid.
- e) Dry the dryer element.
- f) Reassemble the dryer element into its housing.
- g) Test the assembled dryer for leaks and flow.

Procedures

A. General Guidelines

1. **IMPORTANT: Operator must wear gloves at all times when handling Nafion tubing to protect the tubing from contamination with skin oils.**
2. The work area should be kept clean and orderly.



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3. Food and drink should be kept away from the work area.
4. Although Nafion is not hazardous, the nature of any sample residues within the dryer is unknown. Harsh solvent(s) are used to clean the Nafion tubing. Wear proper protective equipment, including at a minimum protective eyewear and rubber gloves. If acids are used to clean the tubing, a labcoat or coverall is recommended.

B. Disassembly of the PD-Series Dryer

1. Disconnect the sample inlet and sample outlet lines from the dryer. If adequate access to the dryer is available, it is unnecessary to disconnect the purge inlet and outlet lines.
2. Remove the front nut (end fitting) from each end of the dryer. Take care not to turn the coupling (middle fitting with connection for purge gas) while removing front nut, as this may twist the Nafion tubing element inside.
3. Wearing rubber gloves, grasp the end of the Nafion tubing epoxy header at one end of the dryer and pull it out sufficiently to expose the two O-rings on it.
4. Remove the O-rings. Repeat this process and remove the O-rings from the other end of the dryer.
5. Slide the Nafion tubing element (bundle) out of the housing.

C. Inspection and Preliminary Cleaning of the PD-Series Dryer Element

1. Visually inspect the Nafion tubing element. Look for discoloration of the Nafion.
 - a) Nafion tubing will discolor over time in normal operation, turning yellow, then progressively darker. This is normal, and does not seriously affect performance. Completely black Nafion is a sign of overheating during operation.
 - b) Other colors (red for copper, green or blue for stainless steel, etc.) indicate contamination with salts from corroded metal upstream from the dryer.
 - c) Solid residues may be deposited within the dryer. Very dark residues are likely oils or tars (residues of organic compounds). Waxy yellow solids may be an indication of ammonium salts (commonly formed when ammonia is injected into stack gas exhausts as part of the DeNO_x process).
 - d) High-boiling liquid residues may be deposited within the dryer. Dark oily liquids are likely sulfuric acid residues.
 - e) If tubes are bent or broken, refer to Repair Procedures.
2. Remove any solids or other visible deposits from the outside of the tubing and from the epoxy header.
 - a) Brush or scrub away any solids deposited on sample inlet header.
 - b) Wipe off any dust or liquid residues from the tubing and header with a clean soft cloth.
3. Blow clean, dry air through the sample outlet end of the header to remove any entrained particles.
4. If contamination with oil or organics is suspected, the dryer may be rinsed with a hydrophobic organic solvent. Most standard degreasing solvents are acceptable as long as they do not mix with water (are non-polar). An example would be hexane.
 - a) Rinse the outside of the dryer element tubing with the solvent.
 - b) Force the solvent through the inside of the tubing. This may be accomplished with a plastic squeeze bottle or syringe.

- c) Blow dry air through the dryer element tubing to remove solvent residues and to dry it thoroughly.
- d) If this cleaning is sufficient, follow reassembly and testing instructions below.

D. Thorough Cleaning (Acid Wash) of the PD-Series Dryer Element

If solid residues are present, the dryer should be washed with acid to remove the residues and to recondition the Nafion (restore it to its original, sulfonic acid form).

1. Prepare a bath of 10% nitric acid (HNO_3) in a suitable container. The container should withstand exposure to nitric acid and heating.
2. Immerse the Nafion tubing into the nitric acid bath, taking care not to wet the epoxy headers. About 1.5 inches (4cm) of tubing as well as the header should remain out of the acid.
3. Do not sharply bend the Nafion tubing during this cleaning process. If the bath is large enough to permit the tubing to flex gradually, the headers may be draped over the edge of the container. Alternately, the dryer element may be suspended over the bath with the headers clamped vertically.
4. A magnetic stirring hotplate should be used if available. If not, the acid container should be heated in some fashion and stirred periodically.

CAUTION: Heating of the acid bath will generate noxious fumes. The cleaning process should be performed in a fume hood or very well ventilated area. Covering the container with a loose plastic lid will minimize the fumes.

CAUTION: Rubber gloves and protective eyewear must be worn. Nitric acid at this concentration will stain and/or irritate the skin and injure the eyes if splashed.

5. Heat the acid bath to 70° to 80°C, and soak the Nafion dryer element for one hour with occasional stirring (a magnetic stirring bar set on low if possible).
6. Remove the dryer from the acid bath. The acid bath may be covered and reused until it shows significant discoloration.

E. Rinsing the PD-Series Dryer Element

1. Rinse the outside of the tubing and of the headers with deionized (DI) water at room temperature to remove the nitric acid residues.

CAUTION: Rubber gloves and protective eyewear must be worn. Nitric acid at this concentration will stain and/or irritate the skin and injure the eyes if splashed.

2. Briefly force DI water through the inside of the tubing. This may be accomplished with a plastic squeeze bottle or syringe. Do not immerse the headers of the dryer element in water or excessively wet the tubing inside the headers. Nafion tubing swells when exposed to water. If the tubing inside the epoxy headers becomes too wet, it may swell sufficiently to crack the rigid epoxy header.

CAUTION: Deionized water must be used. Nafion is a cationic exchange material, and when exposed to liquid water will exchange the hydrogen from the sulfonic acid with any cations in the water. The acid treatment has just reversed this process. Do not then immediately remove the hydrogen, which will seriously degrade the dryer performance.

CAUTION: Rubber gloves and protective eyewear must be worn. Nitric acid at this concentration will stain and/or irritate the skin and injure the eyes if splashed.

F. Drying the PD-Series Dryer Element

1. Blow clean, dry air through the dryer element to remove any residual water.
 - a) Begin blowing dry air through the dryer element quickly after wetting the element. This will minimize the risk of damage to the dryer due to swelling of the tubing.
 - b) Continue blowing dry air through the element for several minutes. This will reduce any swelling and restore the dryer to normal size (length).

G. Reassembly of the PD-Series Dryer

1. Wearing rubber gloves, insert the dryer element into its housing.
2. Install the two O-rings onto the header at one end of the dryer element. The smaller O-ring is installed first.
3. Push the header with the O-rings installed into the housing until the second header appears at the other end of the housing.
4. Grasp the end of the second header, pull it out slightly to expose it, and install the two O-rings on this end.
5. Center the element in the housing with the O-rings equally spaced from the ends of each header.
6. Install the front nut onto each end of the housing and tighten, taking care not to turn the coupling while tightening the front nut.
7. Connect the sample inlet and outlet lines.
8. If the purge was disconnected and the dryer was removed from its mounting bracket, connect the purge lines and the mounting bracket. Note that the purge inlet is at the opposite end from the sample inlet.

H. Testing of the Assembled PD-Series Dryer for Leaks and Flow.

1. After reassembly, the dryer should be tested for leaks and for flow. Please refer to Procedures # PD-006, PD-010, or PD-010A as appropriate.

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