# Q Series

## Nitrogen Purge Cooler Accessory for DMA

# Getting Started Guide

Revision B Issued September 2014

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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.

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# Introduction

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## Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

**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.**

**MISE EN GARDE: UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.**

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

**Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.**

## Safety

**WARNING: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.**

**AVERTISSEMENT: L'utilisateur de cet instrument est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.**

### *Instrument Symbols*

The following labels are displayed on the Nitrogen Purge Cooler for your protection:

| Symbol | Explanation |
|---|---|
|  | This symbol indicates that you should read this Getting Started Guide for important safety information. This guide contains important warnings and cautions related to the installation, operation, and safety of the NPC.<br><br>Ce symbole indique que vous devez lire entièrement ce guide de démarrage pour obtenir d'importantes informations relatives à sécurité. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité du système NPC. |

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DMA Nitrogen Purge Cooler Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

## *Chemical Safety*

**WARNING: Do not use hydrogen or any other explosive gas in the NPC.**

**AVERTISSEMENT: N'utilisez pas d'hydrogène ou tout autre gaz explosif dans le NPC.**

**WARNING: If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.**

**AVERTISSEMENT: Si vous utilisez des échantillons qui émettent des gaz nocifs, ventilez les gaz en plaçant l'instrument près d'un échappement.**

**WARNING: Air is not to be used as the purge gas with this cooler. Oxygen will be condensed into liquid and pushed into the DMA furnace.**

**AVERTISSEMENT: l' Air ne doit pas être utilisé les gaz de drainage comme avec ce refroidisseur. L'Oxygène se condensera en liquide et sera poussé dans le four DMA**

**WARNING: MAY CAUSE SEVERE FROSTBITE. Can cause severe frostbite to the eyes and skin. Do not touch frosted pipes or fittings.**

**AVERTISSEMENT: Peut causer de graves gelures. Peut causer des gelures graves aux yeux et à la peau. Ne pas toucher les tuyaux ou les raccords givrés.**

**WARNING: Do not overfill the Dewar. Leave an inch of space from the top of the Dewar when filling. Liquid nitrogen may boil over the sides when the Cooling Gas is turned on.**

**AVERTISSEMENT: Ne surchargez pas le Dewar. Laisser un espace d'un pouce à partir du haut du Dewar lors du remplissage.**

## Handling Liquid Nitrogen

This cooling accessory uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature (–196°C [–321°F)]), liquid nitrogen will burn the skin. When you work with liquid nitrogen, use the following precautions:

**WARNING: Liquid nitrogen boils rapidly when exposed to room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.**

**AVERTISSEMENT: L'azote liquide bout rapidement lorsqu'il est exposé à la température ambiante. Assurez-vous que les zones où l'azote liquide est utilisé sont bien aérées pour éviter le déplacement de l'oxygène dans l'air.**

1. Wear a face shield, gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.
2. Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.
3. The purity of liquid nitrogen decreases when exposed to air. If the liquid in a container has been open to the atmosphere for a prolonged period, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

**NOTE**: Using the NPC poses all of the same risks as with any open Dewar of cryogenic liquid. Safe practices concerning the filling and transporting of small Dewars and use of Personal Protective Equipment is necessary.

The asphyxiant warning below applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes used where liquid nitrogen is in use.

**WARNING:**
**Potential Asphyxiant**

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent the Nitrogen Purge Cooler (NPC) in confined spaces.

Do not enter confined spaces where nitrogen gas may be present unless the area is well ventilated.

**AVERTISSEMENT:**
**Asphyxiant Potentiel**

L'azote liquide peut provoquer un étouffement rapide sans prévenir.

Entreposez-le et utilisez-le dans une zone bien aérée.

N'aérez pas le NPC dans des espaces confinés.

N'entrez pas dans des espaces confinés où l'azote gazeux peut être présent à moins que la zone soit bien aérée.

# Table of Contents

# *Chapter 1:*

# Introducing the Nitrogen Purge Cooler

## *Overview*

The Nitrogen Purge Cooler (NPC), is an optional accessory for extending the temperature range of the Q800 DMA standard furnace to -160°C. The NPC consists of a 2.5 L Dewar flask that contains a copper coil tube. The furnace is cooled by purging nitrogen gas through the copper coil immersed in the liquid nitrogen-filled Dewar flask. The NPC requires a nitrogen gas supply (25 to 120 psi) at a flow rate of 30 LPM, and access to a supply of liquid nitrogen and equipment necessary for safe handling, transportation, and pouring of the liquid.

**NOTE**: The NPC is for crash cooling and controlled heating only. Controlled cooling rates are possible using the Gas Cooling Accessory (GCA), or Air Chiller System (ACS-3).

## *System Components*

The NPC Accessory has the following major hardware components:

- A Dewar flask with insulated lid, capable of holding approximately 2.5 L of liquid nitrogen, and with a precise length of copper tubing to adequately chill the nitrogen purge gas.
- A length of 1/8” OD tubing that connects the NPC to the Cooling Gas Outlet fitting on the rear of the DMA.

# *Accessory Specifications*

The tables below detail the NPC Accessory technical specifications.

### Table 1: NPC Accessory Characteristics

| | |
|---|---|
| **Dimensions of Dewar flask** | Height: 15 in.<br>Width: 12 in.<br>Depth: 7 in.<br>Volume: 2.5 L |
| **Weight of Dewar flask** | 6 lbs (without liquid nitrogen) |

### Table 2: Accessory Cooling Gas Requirements

| | |
|---|---|
| **Gas** | Nitrogen ONLY |
| **Pressure** | 25–120 psi |
| **Flow Rate** | 30 LPM |

### Table 3: Accessory Operating Environmental Conditions

| | |
|---|---|
| **Operating altitude** | 2000 meters maximum |
| **Relative humidity** | 5% to 80% RH from 15°C to 31°C, decreasing to 66% RH at 35°C (non-condensing) |

## Table 4: Accessory Performance Specifications

| | |
|---|---|
| **Lowest temperature** | -160°C |
| **Time to reach low temperature** | 40 min |
| **Time to reach -150°C** | 35 min |
| **Heating rate:**<br>**Minimum**[a] | 3°C/min |

a. With NPC off and dependent on ambient temperature.

# *Chapter 2:*

# Installing the Nitrogen Purge Cooler

## *Unpacking/Repacking the NPC*

The instructions needed to unpack and repack the accessory are found as separate unpacking instructions in the shipping box and in the online documentation associated with the instrument control software. Be sure to read and perform the unpacking instructions prior to performing any procedures in this chapter.

Retain all of the shipping hardware and boxes from the accessory in the event you wish to repack and ship your accessory.

## *Preparing the Accessory*

Before shipment, the NPC is inspected so that it is ready for operation upon proper installation. Only limited instructions are given in this manual; consult the online documentation for additional information. Installation involves the following procedures:

- Inspecting the accessory for shipping damage and missing parts
- Connecting the NPC tubes and fittings
- Filling the Dewar

**CAUTION: To avoid mistakes, read this entire chapter before you begin installation.**

**MISE EN GARDE: Pour éviter de commettre des erreurs, lisez tout le chapitre avant de commencer l'installation.**

## Inspecting the System

When you receive the NPC Accessory, look over the accessory and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the accessory is damaged, notify the carrier and TA Instruments immediately.
- If the accessory is intact but parts are missing, contact TA Instruments.

## Choosing a Location

Choose a location for the accessory using the following guidelines. The NPC Accessory should be:

### *In*

- A temperature-controlled area.Temperatures should be in the range of 20–35°C.
- A clean environment, preferably on the ground floor in the building.
- An area with ample working and ventilation space.

### *On*

- A stable work surface.

### *Near*

- Your Q Series DMA.

### *Away from*

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Noisy or mechanical vibrations.
- High traffic areas, where constant movements from passing personnel could create air currents or mechanical disturbances.

# *Connecting the NPC to Your Q Series DMA*

To connect the NPC, access the rear panel of the DMA and follow the instructions below:

**NOTE**: Remove the foam insulation from the Dewar before making connections and filling the Dewar with nitrogen.

**1** Place the Dewar next to the DMA.

**2** Disconnect the GCA tube from the DMA.

**Figure 1** Disconnect the GCA tube.

**3** Connect the copper tubing to the GCA port on the DMA.

**Figure 2** Connect copper tubing to the DMA.

**4** Fit the tube insulation piece over the GCA port connection.

**Figure 3** Tube insulation piece covering the GCA connection.

**5** Connect nitrogen purge gas to the Cooling Gas Inlet port (see Figure 4 below) on the DMA. Inlet pressure should be between 25 and 120 psi. Use nitrogen gas ONLY.

**6** Remove the silicone tubing from the barb fitting on the Air Cool Outlet port, then remove the barb fitting.

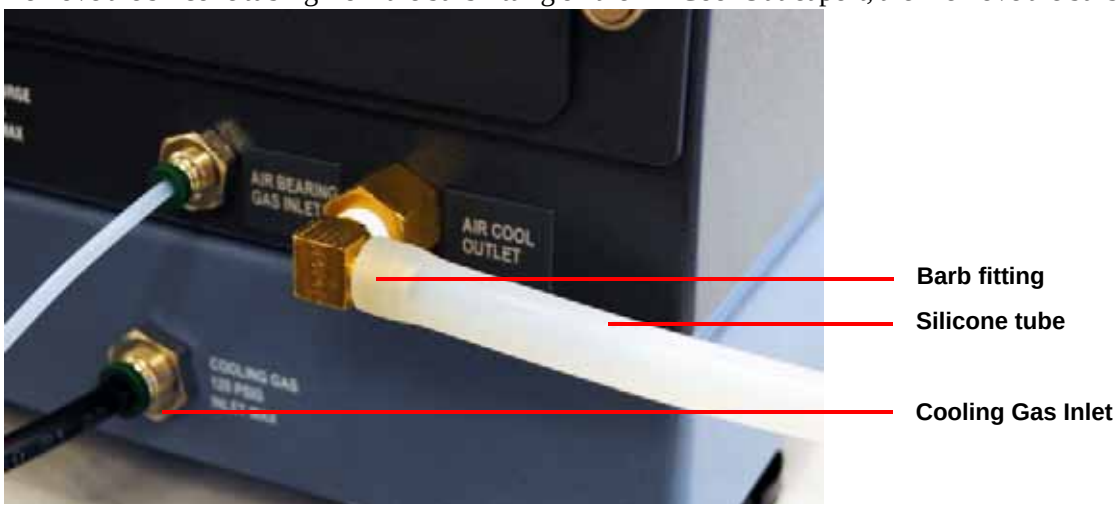


**Figure 4** Barb fitting with silicone tubing attached to the Air Cool Outlet port.

**7** Place the Cooling Gas Outlet label directly over the Air Cool Outlet label so that “Air Cool Outlet” is completely obstructed.

**8** Attach the quick-disconnect elbow to the Cooling Gas Outlet port.


**Figure 5** Quick-disconnect elbow fitting attached to Cooling Gas Outlet port.

**9** Insert the empty end of the 1/8” black tubing into the end of the elbow. Then screw the nut attached to the end of the tube to the Swagelok fitting on the copper tubing.

**Figure 6** Black tubing (left) connected to the copper tubing in the Dewar (right).

**NOTE**: The 20” length of 1/8” tubing is critical. Do not shorten this tube. Shortening this tube can cause an excessive amount of nitrogen to be condensed into liquid and pushed into the furnace.

**10** Proceed to the next section to set up the accessory in Thermal Advantage. Do not fill the Dewar until the next section has been completed.

# *Setting up the NPC Accessory using Instrument Control Software*

**CAUTION: Operation of this accessory is intended for the QNX-based DMA Q800 with instrument software version 21.2.73 or higher only.**

**ATTENTION: L'utilisation de cet accessoire est compatible avec le DMA Q800 uniquement en base QNX et version de logiciel de l'instrument 21.2.73 ou supérieur.**

After successfully installing the NPC Accessory, access the DMA Thermal Advantage Software and follow the instructions below.

**1** Select **Control > Select Accessory**

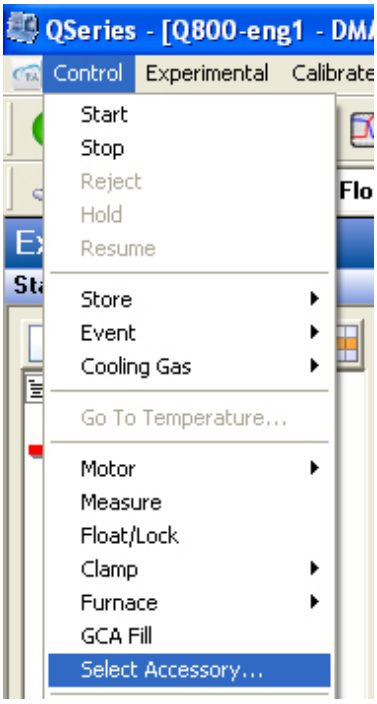


**Figure 7** Select Accessory.

**2** In the DMA Cooler Selection dialog box, select **NPC**.

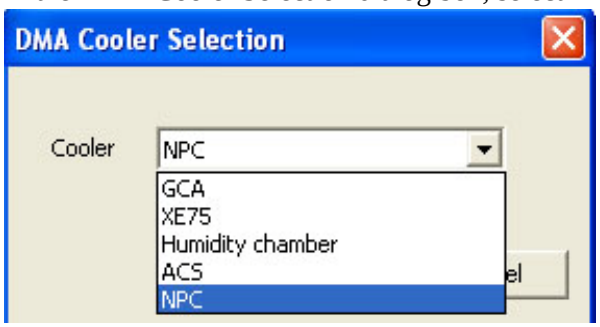


**Figure 8** Select NPC.

**3** Proceed to Filling the Dewar.

# *Filling the Dewar*

Before the Dewar can be filled with liquid nitrogen, the copper tube must be purged with dry gas. This is necessary to prevent moisture from freezing in the copper tube when liquid nitrogen is added to the Dewar; the frozen moisture blocks the flow of the cooling gas.

**1** Make sure the furnace is closed.

**2** In Thermal Advantage, select **Cooling Gas** > **On** from the **Control** tab.

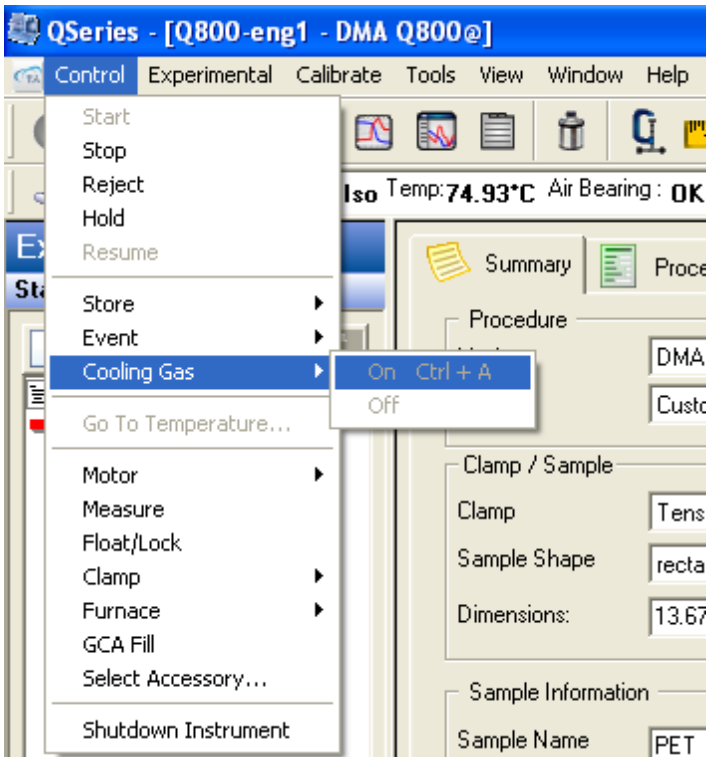


**Figure 9** Turn on Cooling Gas in Thermal Advantage software.

**3** Allow the dry gas to purge through the copper tube for at least 20 minutes, or until the copper tube is dry.

**4** Turn the Cooling Gas off (the same way it was turned on in the software).

**5** Fill the Dewar with liquid nitrogen.

**WARNING: Do not overfill the Dewar. Liquid nitrogen may boil over the sides when the Cooling Gas is turned on.**

**AVERTISSEMENT: Ne pas trop remplir le Dewar. L'azote liquide peut avoir une ébullition sur les côtés lorsque le gaz de refroidissement est activé.**

# *Chapter 3:*

# Use and Maintenance

## *Using the NPC Accessory*

All of your NPC Accessory experiments will follow the same general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. See the instrument control online help for instructions on performing these actions.

- Filling the Dewar
- Selecting and preparing the sample
- Creating or choosing a test procedure and entering experiment information through the TA Instruments instrument control software
- Loading the prepared sample
- Starting the experiment

Please note the following when performing an experiment:

- One full Dewar (approximately 2.5 L of liquid nitrogen) will typically be consumed for each experiment.
- A full Dewar is sufficient for performing the following method when the starting temperature is 40°C:
  - External Event: ON
  - Equilibrate: -150°C
  - Isothermal: 5 minutes
  - External Event: OFF
- When the NPC is connected to a bottle of dry, compressed nitrogen at approximately 2000 psi, seven complete experiments as shown above can be performed.

### Before You Begin

Before using the NPC Accessory, ensure that the DMA Q800 Dynamic Mechanical Analyzer is installed properly. Also make sure you have:

- Connected the NPC
- Powered on the DMA
- Filled the Dewar as necessary
- Specified the NPC in the instrument control software
- Become familiar with controller operations

It is a good idea to purge the copper coil with dry nitrogen just prior to filling the Dewar with liquid nitrogen. Select **Control** > **Cooling Gas** > **On** to purge the coil with dry nitrogen. After a few minutes, select **Control** > **Cooling Gas** > **Off** to turn the purge gas off again.

# *Running an NPC Experiment*

## Creating an Experimental Procedure

**1** From the **Procedure** tab, select the test you want to run from the **Test** drop-down menu

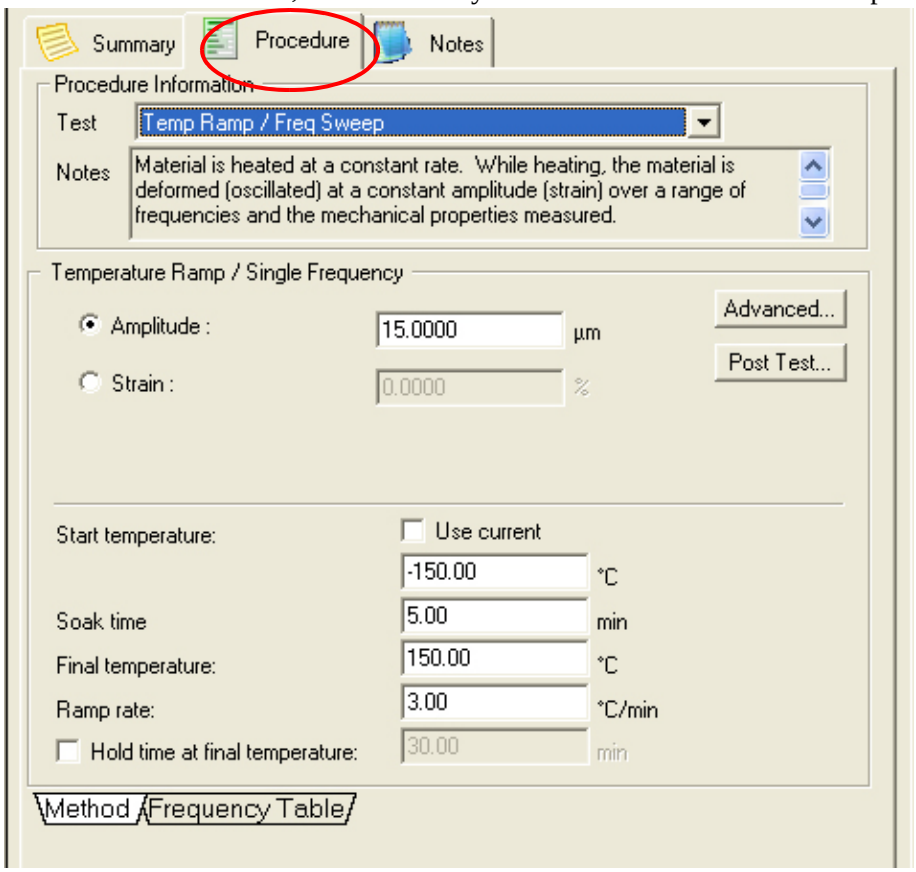


**Figure 10** Procedure tab > Test menu.

**2** Change the ramp/frequency and temperature fields as needed.

**3** The experiment steps display on the right in the **Running Segment Description** section. Click **Apply** when you have the experiment set up to your satisfaction.

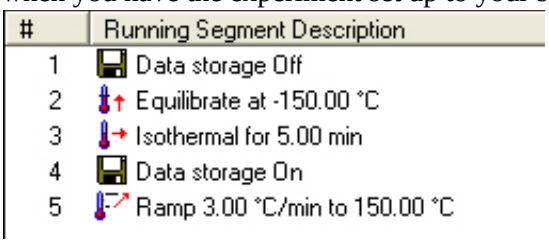


**Figure 11** Experiment steps.

## Editing an Experiment

**1** In the **Procedure** tab, select **Custom** from the **Test** drop-down menu, and then click **Editor**.

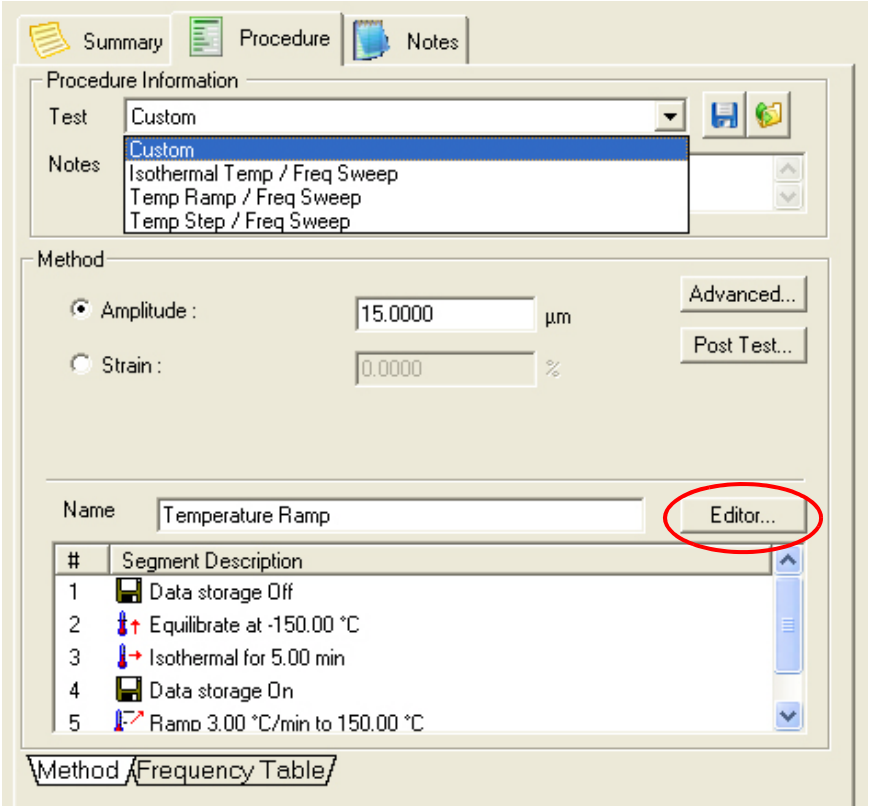


**Figure 12** Custom > Editor.

**2** A **Method** dialog box displays. Drag experiment steps from the Segment list and drop them into the Segment Description section where needed. Click **OK** when finished.

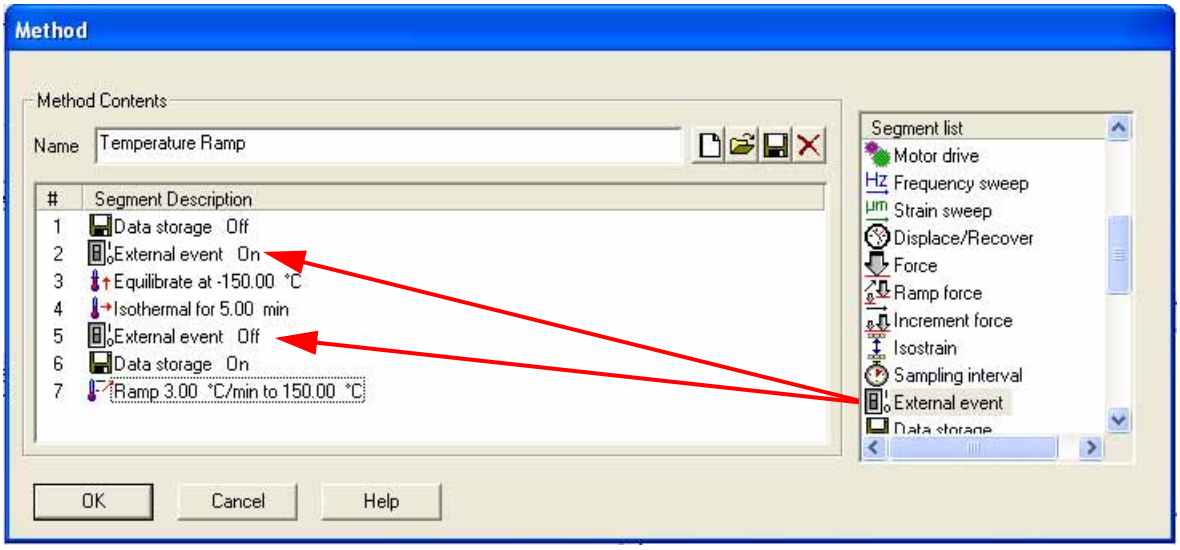


**Figure 13** Edit the experiment steps.

**NOTE**: The segment “External Event On/Off” refers to turning the Nitrogen Purge Cooler on or off.

# *Maintaining the Accessory*

The primary maintenance procedures described in this section are the customer’s responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel.

## Cleaning the Accessory

To clean the NPC Accessory, wipe down the exterior of the Dewar with a damp, soft cloth.

**CAUTION: Do not use harsh chemicals, abrasive cleansers, steel wool, or any rough materials to clean the cabinet, as you may scratch the surface and degrade its properties.**

**MISE EN GARDE: N'utilisez pas de produits chimiques agressifs, de nettoyants abrasifs, de la laine d'acier ou tout autre matériau rugueux pour nettoyer l'armoire, car vous pourriez égratigner sa surface et dégrader ses propriétés.**

# *Replacement Parts*

**Table 5: Replacement Parts for the NPC Accessory**

| Part Number | Description |
|---|---|
| 986316.901 | Insulation Replacement Kit NPC |