IOWA STATE UNIVERSITY
Chemical Instrumentation Facility

Netzsch TGA/DSC—IR/MS Short Manual

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The Netzsch STA449 F1 is a combination Differential Thermal Analysis/Scanning Calorimetry instrument coupled to an FTIR and Mass Sensitive Detector for evolved gas analysis.

Purpose

This document is meant to be a simple-to-read refresher outlining the setup and use of the Netzsch TGA/DSC-IR/MS instrument. It assumes the reader has familiarity with the setup and does not go in to intricate details, but will mention each step required to walk in, operate the instrument, and collect data while being easy to read. *This is not meant to be a training document.*

Getting Started

For time reservations of this instrument, please use the Google Calendar functionality of your CyMail account. You will be added to the calendar once you complete training.

To begin a TGA measurement, log in to the *LockScreen* program using your unique password which unlocks the screen. All of the programs necessary for a TGA measurement are in the pool

of shortcuts at the top of the main monitor. The first step is to check the status of the furnace and set up the correct gases. Start by launching the *STA449 F1* program.

To check the status of plumbed gases, open the *MFC Extended Gases* window (two gas cylinders). After setting up the purge and protective supply tanks and valves for what you need, modify the software settings to reflect these changes. A total flow rate of ~60 mL is recommended, with at least 20 mL/min on the protective line. Also, the protective line should always be UHP gas, whether using air, Ar, or N_2.

To check the status of the furnace, press the *ASC Test* (wrench with 3 dots) button. This will load a window full of autosampler commands. Verify the furnace status and sample carrier by selecting "*Move to sample carrier change position*". This will raise the furnace and spin it out of the way, allowing for viewing the sample carrier. Verify the sample carrier is clean, then the furnace may be closed again.

At this point, close *STA449F1*.

Building the Macro

Now launch *Autosampler* to begin constructing the main macro program. First, verify the configuration with *File->Configuration*. Verify plumbed gases, and decide whether to have the instrument balance mass the crucibles and samples, or input your own masses. Always leave the FTIR and Quadstar trigger setups *ON*—data collection from the gas analysis instruments can be allowed or denied later in the programming.

Once the configuration is set, select *New Macro* from the menu, and another window will open. Enter in your three digit initials code followed by the date and –mac (i.e. bwb-01012020-mac) and your network id as the creator. This is where autosampler position data for the crucibles you want to run will be input. Select *New* to add a step in the analysis macro. This will pop up the step definition window, where all identifiers and the temperature/gases program will be entered.

In the new window, the configuration will be showed again for verification. Proceed to the *Position* tab to define the crucible type (empty or filled) and continue to *Step Data*. This is where the directory, filename, sample name, and sample ID must be filled in. Our naming conventions are as follows:

> Current Directory: D:\Data\universityID\
> Data Filename: *xyz-date-position* (i.e. bwb-01012020-0)
> Sample Name: anything you want
> Sample ID: any other identifier (notebook page,etc) or same as sample name.

Once these are finished, proceed to *Measurement* to set up the furnace conditions. Unless you are repeating a previously set up experiment exactly (down to the gases, flow rate, etc), select *New Configuration*. This will load another window, again showing the configuration.

Navigate to the *Header* tab to input some parameters, including the left side data block (Lab, project, etc). Importantly, both the temperature and sensitivity calibrations need to be selected based on crucible type, carrier gas, and ramp rate. Select the best calibration from the prompt windows. Once the header is complete, move forward to the *Temperature Program*.

The furnace idles at about 38°C, so a good initial temperature is 35-40°C. If you are looking to have pure inert carrier gas in the mass spec signal, it is advised to evacuate+fill the balance cell (protective line) at least once with the carrier gas of choice for the first run, and include a 10 minute isothermal step at the beginning of the run to clear additional air out of the furnace at the start.

The remainder of the temperature program generally consists of one or more dynamic ramp steps to a final temperature, with an optional high temperature isothermal step. Select from the right-hand menu for each step you would like to program, set up the gas flow rates, and check the FTIR box if you are planning to collect IR evolved gas data. *Always leave STC enabled for all steps*. When finished, add a *Final* step which is just a fail safe against the furnace overheating. When the program is complete, press *OK* to send the program back to the step definition window. Once that is done, press *OK* to send all the parameters back to the main maco window.

Now that the first position step is complete, the rest is much easier. Simply *Copy* that step, *Paste* the new step after the previous, and the program will bring up the *Position* tab again. For this step, select *Filled Crucible* since these steps will have samples. Move *Forward* to fill in the data block for the first sample, keeping with the naming conventions. After filling in the names, move forward to verify the temperature program. It will already contain the program from before, so select *OK* to finish the step.

To finish out the magazine for as many samples as you need to run, copy and paste this second step repeatedly, making sure to update the data block for each new sample. Once this is all finished, save the macro in the default location.

Mass Determinations and Starting the Run

Once the macro is set, all further commands will be called from the *Macro* tab. Select *Determine Crucible Masses* to collect mass information on the crucibles after loading the appropriate number of crucibles in the autosampler tray for your experiment. The software will get ready to begin, but requires one more input to start—it asks the operator to verify the furnace status (which is why we looked in the beginning). Press *OK* to begin the mass determinations.

Once the crucible masses are determined, the instrument will show a small window saying the masses were determined and present a dialog box showing the values. Press *OK* to close that window and then load 2-5 mg of sample into each sample crucible (*At low temperatures or under certain conditions more mass may be loaded but this is a good rule of thumb*). Then

return the crucibles to the autosampler tray and select *Macro->Determine Sample Masses*. Once again the furnace status must be verified prior to the mass determination.

Note: While the sample mass determinations are determined you may fill the liquid nitrogen dewar for the TGA-IR detector if you are planning to collect FTIR data.

A small window and report will be generated after the sample masses are determined. Just in case of problems with the sample runs, save the macro after all the masses are determined so all the information is saved.

Now the macro is ready to run. Select *Macro->Run Magazine* to begin. A verification message pops up, showing the post-run conditions which should all be correct and can be left as-is. Then another window with the steps that will be run will be shown in green, which is helpful in the event of an unlikely instrument error. Press *OK* to continue and the *STA449F1* program will launch. When presented with the small "For standard ASC runs" dialog, **STOP!**

Now the other instrument software needs to be launched for collecting a combination of FTIR and MS data. If FTIR data is desired, launch *OPUS* (password: OPUS) and select the top left beaker icon, verifying the TGA method is loaded and select *Check Signal* to see the amplitude of the interferogram is above ~17000 counts or so. Now the FTIR is ready for about 5 hours of data collection before the liquid nitrogen must be refilled.

For mass spec data, launch *Measure+Sequencer* from the top desktop icons, which will initialize the Aeolos mass sensitive detector and collect some vacuum data on the source. After ~30 seconds or so, a small window on the left side of the left monitor will load, and for most data sets select *Scan Bargraph*. Another dialog box will load and ask for your parameters file, which you can use your own parameter set or use mine (bwb-001) for 10-200 m/z at standard resolution and filament current. Once this is selected, the measurement window will show at right, and a small green light and "Waiting for trigger" message will appear in the lower left. Now the secondary data streams are ready, and you may select *OK* from the "For standard ASC runs" dialog.

The instrument will start after some delays and FTIR background (if applicable), and then the temperature program will start. At this point the instrument is on autopilot and no further input is necessary until all the data runs are complete. The only thing to maintain now is the FTIR liquid nitrogen level, which should be filled every 5 hours from when it was initially filled.

Clean up and Shut Down

Once the runs have completed, all software windows should be closed. If mass spec data was collected, find it in D:\DATA\Mass_Spec_Data and copy it to your own folder. Remove crucibles, clean the solids from them, and place them on the PulseTA in the dirty crucible pile.

Once finished, use the small lower right *Logout* button to close your instrument session.