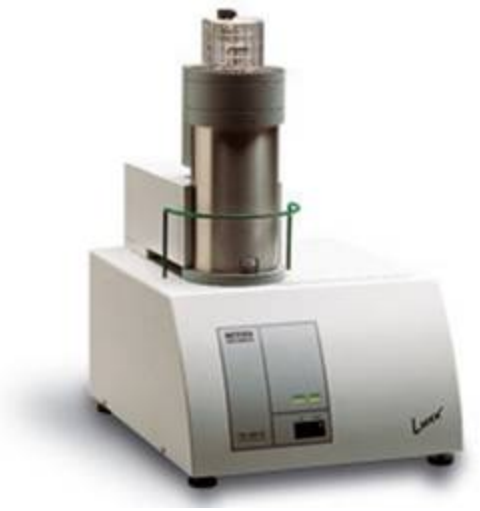
NETZSCH (DSC/TGA/DTA)

Rev. 8/1/2013

Description



The following are standard operating procedures for the Netzsch Simultaneous Thermal Analyzer STA409 PG. This instrument is used for various thermo-analytical measurements such as DSC, TGA, and DTA. Sample masses often vary from approximately 2-20 mg, depending on operation mode. Sample types vary and may include loose powders, thin pellets, metal chunks, and others.

Before starting

1. **Be sure to be wearing PPE (personal protective equipment) such as gloves, eye protection, and lab coat upon entering the lab.**
2. Know which operation mode is desired and insert appropriate sample carrier. **Seek help before attempting to remove/replace carriers!**
3. Ensure that instrument is on. The "POWER" light should be lit in the front of the instrument.
4. Check gas lines connected to Room 109. Make sure you can see appropriate flow settings on rotameter.
5. Make sure that argon gas is always flowing the instrument (usually set to 50 mL/min) to protect the instrument's furnace.
6. Never open/touch furnace if the temperature exceeds 50 °C.
7. Do not place hands under moving furnace. Pinch Hazard!
8. Do not lean on the instrument.

Instrument Operation

1. Using tweezers, take appropriate crucible and weigh it in mass balance (located on countertop).
2. Tare the mass of the crucible and remove crucible from the balance.
    a. For TGA and DTA crucibles, place crucibles on crucible holder (located inside if balance chamber) and tare holder mass along with crucible.
3. Place crucible on counter and carefully load powder into the crucible, place into balance again, and make note of powder mass.
    a. For crucibles with walls like for DSC and DTA operation, ensure that no powder sits at the top rim of the crucibles.
4. If using a lid, carefully place lid on top of crucible (lip side down).
5. On the instrument, lift furnace cover by pressing the "up" button together with the safety button (located at the front and right side of the instrument, respectively). This activates the hoist located in the back of the chamber.
6. Using tweezers, carefully place sample crucible onto the carrier. For carriers requiring reference crucibles, place similar empty crucible/lid in reference side of carrier.
7. To lower the furnace cover, press the "down" button together with the safety button (located at the front and right side of the instrument, respectively).
8. Make sure the two red valves for the gas lines in the back of the instrument are closed.
9. Close the evacuation /gas outlet valve located the top of the instrument.

10. Begin procedure of evacuating air/filling chamber with Argon (repeat 2-3 times).
    a. Turn on pump located on floor to the left of the instrument.
    b. Partially open the vacuum valve located in the back left side of the instrument. Keep opening this valve little by little until the pressure (read on gauge also in the back, left side of instrument) reads the max negative pressure. User should leave the vacuum to stabilize until a buzzing sound can be heard. This ensures that most of the air is evacuated.

       WARNING: Abruptly the vacuum valve all the way will disturb the sample crucible and can cause powder/sample to fall out and contaminate the instrument.

    c. Once all air has been evacuated, close vacuum valve and slowly/partially open the Argon gas valve located in the back left side of the instrument to fill the chamber with Argon. User should see the gauge pressure increase to zero again. At zero, close the Argon valve and begin evacuation again.
11. Evacuation of air and filling with Argon should be repeated 2-3 times. On the last filling of Argon, one should fill a little more than zero.
12. Slowly open appropriate gas valves for use in experiment. (red valves in the back of the instrument)
13. Open the gas outlet/evacuation valve at the top of the instrument.
14. Begin setting up the software settings and input desired temperature program.
15. After setting up software, press start. This should turn on the light above "ON" at the front of the instrument. Remember, the "POWER" light should already be lit.
16. After pressing start, a message pops up about the temperature not yet being stabilized. Press OK. To start measurement right away, press start again. Not pressing start will allow instrument to stabilize the temperature. This lasts for 5 hours then the measurement will start automatically.

After measurement (extraction of the samples from the instrument)

Samples that have undergone heating should either be properly disposed of in powder waste containers available in the lab.