

Thermal Gravimetric Analyzer (TGA)

Instrument must meet or exceed the following minimum specifications:

Weight capacity of 750 mg (maximum sample weight independent of pan)

Dynamic weighing range of ± 100 mg (independent of pan, defined as the maximum measurable weight change)

Weighing precision of $\pm 0.01\%$, (defined as the standard deviation of at least 10 measurements of a 100mg standard weight, with removing and replacing the sample between each measurement)

Dynamic baseline drift (50 to 1,000 °C): <10 μg , with platinum pans (defined as the maximum deviation from the smallest measured weight to the largest measured weight of an empty platinum pan, while being heated at $20^\circ\text{C}/\text{min}$ in flowing nitrogen atmosphere (without any blank subtraction applied))

Baseline linearity (50 to 1,000°C): <1 μg , with platinum pans (defined as the average absolute deviation from a best fit linear regression of a baseline scan without any smoothing or blank subtraction applied)

Signal resolution: $0.002\mu\text{g}$ (defined as the smallest measurable difference between two adjacent values)

Sensitivity: <0.1 μg (1 ppm) (defined as 3X the average rms noise over the temperature range 50 to 1,000°C)

Temperature range: ambient to 1200 °C (defined as the measured temperature at the sample thermocouple (not furnace temperature or programmed temperature))

Temperature accuracy: $\pm 1^\circ\text{C}$ (defined as the standard deviation of the measured error (at least 10 replicate runs after temperature calibration) of the onset temperature of a nickel Curie Point measured at $10^\circ\text{C}/\text{min}$, removing and replacing the sample between each run)

Dynamic temperature precision: $\pm 1^\circ\text{C}$ (defined as the standard deviation of the measured Curie Point temperature of at least 10 nickel runs, removing and replacing the sample in between each run)

Isothermal temperature precision: $\pm 0.1^\circ\text{C}$

Linear heating rates: 0.1 to 500 °C/min in $0.01^\circ\text{C}/\text{min}$ increments

Ballistic heating rates: > 2000 °C/min

Furnace cooling must be forced air 1000°C to 35°C in < 10 min.

Furnace must possess built in electromagnetic coil for automated temperature calibration

Purge gas delivery control must accommodate up to four simultaneously installed gases. This capability must be incorporated into the instrument (i.e. shall not be a separate unit).

Purge gas flow rate must be programmable within operating software, and deliverable as a saved signal in the data file.

Gas delivery control must also allow for automated switching between the gases during an experiment, as well as blending of binary mixtures of gases in controllable ratios.

Must contain an auto-sampler capable of holding at least 25 samples with automated pan punching

So as to isolate vibration producing components from the TGA balance, the TGA must be comprised of separate cabinets, one containing the TGA transducer, furnace, gas delivery components and associated electronics and the second containing the power supplies and supporting electronics.

Electronics cabinet must be upgradeable to support two separate thermal analysis transducer modules simultaneously.

Must include high-sensitivity, temperature-controlled thermobalance as described below.

Must include IR-radiation furnace, as described below.

TGA must include evolved gas analysis (EGA) capability without resorting to a second furnace.

TGA must be easily interfaced to a Mass Spectrometer or FTIR, and shall accommodate a heated purge gas outlet to minimize condensation. The relative temperature of the heated outlet must be software controlled.

TGA must include touch screen capable of recalling and running preprogrammed methods, loading, taring, and monitoring running experiment.

TGA must communicate with computer/controller through Ethernet BUS.

Data files contain measured sample temperature, not calculated temperature to allow user to know what temperature the sample is actually at during different heating rate experiments and makes for accurate and precise transition temperatures.

Must demonstrate the ability to continuously measure sample weight loss of up to 100 mg.

Must employ single thermocouple design with continuous use of the measured sample temperature to control furnace in order to minimize thermal lag.

Must employ horizontal purge gas flow to minimize buoyancy effects from purge gas and for direct output to off gas analysis.

Must employ push button automatic loading and unloading of sample pan (so that operator does not have to hang pan on wire in normal use).

Must demonstrate ability to have up to five points for temperature calibration to provide greater temperature accuracy over wide temperature ranges.

Must be capable of Curie Point temperature calibration.

Must include operating software which allows for the instrument to be fully calibrated and verified automatically, without the need for operator presence.

Calibrations must include weight and temperature.

The data analysis software should be unkeyed, to allow for unlimited installations within one site.

The data file format should easily allow sharing/transfer of data files as individual electronic documents, which are readable by the same data analysis package.

The data analysis program should also include a .pdf generator, for the efficient export of analyzed plots.

Software shall include auto-tare feature to tare each pan unattended.

Instrument must be capable of performing a unique experiment on each sample within one loaded sample tray.

Autosampler must be capable directly punching sealed pans under force control and in a contamination-free manner. (direct piercing by a sharp "awl-like" mechanism does not constitute a contamination-free process.)

System must have the capability of rejecting any pan that is not pierced to prevent a sealed pan being loaded into the TGA furnace.

Shall employ software-driven position calibration routines to ensure reliable performance. Total calibration of position shall take no more than 5 minutes.

Must possess ability to perform Hi-Res TGA measurements (defined as an advanced heater control technology to optimize weight loss resolution using the following three modes. All modes are used to separate overlapping transitions. (1) Dynamic Heating Rate to automatically

and continuously change heating rate as a function of decomposition (sample weight loss), (2) Constant Reaction Rate to automatically and continuously change heating rate to achieve a pre-determined rate of sample decomposition expressed in %/min, (3) Stepwise Isothermal to automatically change from heating to an isothermal hold when pre-selected limits of weight loss in %/min are met.

Ability to abort a test and/or segment in a test when operator specified conditions are met.

Must have local control at the module including experiment start/stop and real-time display of sample temperature and experiment status.

Automatic recording of initial sample weight.

Automated push button taring.

One button push loads pan, raises furnace, tares pan, and unloads pan.

Ability to continue a run when disconnected from the computer/controller.

Ability to use pans up to 250 μ l volume so as to fit larger samples in thermobalance.

Ability to store calibration constants in module memory.

Instrument should be compatible with current Discovery Common Cabinet (P/N: 922000.901) equipped with dual module upgrade kit (P/N: 922303.901).