

## Specifications for a Thermal Analysis System

The Naval Research Laboratory has a requirement for a thermal analysis system, which is to include a Modulated Differential Scanning Calorimeter (DSC) and a Thermogravimetric Analyzer (TGA), along with requisite software to control the system, an accessory to enable sample cooling below room temperature, and a software upgrade to an existing ARES mechanical spectrometer. The software needs to be capable of running the DSC equipment as specified below, and the computer's operating system needs to be compatible with NRL's existing Microsoft Windows XP Pro. The purpose of this equipment will be to support the mission of Navy research into the dynamics of glass-forming liquids, coatings, and high temperature materials. Specifically, this equipment will be used to: 1) quantify the transition temperatures and activation energies for segmental and secondary relaxations in polymers and mixtures; 2) characterize the thermal properties and thermal stability of various resins and coatings; 3) measure startup transients during the flow of viscoelastic materials; 4) detect thermal transitions in nanoscale organometallic materials; and 5) determine pyrolytic organic content in nanoscale organometallic materials. The materials to be analyzed frequently exhibit weak and/or overlapping transitions, which require a flat, repeatable baselines and exceptional thermal resolution in DSC measurements. The system, described here and elsewhere, shall be capable of being operated on the same computer. All specifications shall be verified using publicly available data, including experimental details, which will permit verification of reproducibility and repeatability of the cited results, when a user adequately skilled in the use of the equipment repeats the experiment. The system must meet or exceed the following minimum specifications:

### 1.0 Specifications for the Modulated Differential Scanning Calorimeter (DSC):

- a) **Baseline Curvature:** There shall be no greater than 20  $\mu\text{W}$  of curvature over a temperature range of  $-20^{\circ}\text{C}$  to  $300^{\circ}\text{C}$ , as determined via a linear interpolation of the temperature extremes. This performance must be repeatable and reproducible over at least 10 repeat runs, without exceeding the 20  $\mu\text{W}$  deviation.
- b) **Modulated Temperature DSC Capability:** To study close-lying relaxation peaks in polymeric and molecular glass-forming liquids, the system must be capable of modulated temperature DSC in both the heating, and pseudo-isothermal modes (average isothermal temperature with a superimposed modulation in the temperature). Publicly available data, as described previously, shall be made available which can verify this capability. As part of this capability, the following data shall be available as separate signals recorded during the experiment, with a selectable data sampling frequency of 5 data points per second or greater modulated temperature; heat flow: net/total,  $C_p$  (reversing), and kinetic (nonreversing); phase angle; and heat capacity: complex, in-phase, and out-of-phase.
- c) **Baseline noise:** There shall be no greater than 1  $\mu\text{W}$  baseline noise (peak-to-peak) during a heating or cooling rate of  $1^{\circ}\text{C}/\text{min}$  (without smoothing). This requirement must be achievable over the entire operating temperature range of the equipment. Real data must be made available to document this capability.

- d) **Temperature Range:** Achievable temperature range shall be from a minimum no greater than  $-180^{\circ}\text{C}$ , and a maximum no less than  $550^{\circ}\text{C}$ . Cooling shall be achieved using liquid nitrogen. Cooling rate equal to at least  $5^{\circ}\text{C}$  under control down to  $-160^{\circ}\text{C}$ . Publicly available data, as described previously, shall be made available which can verify this capability
- e) **Purge Gas Control:** Gas drying capability to remove moisture from purge gas.
- f) **Baseline Reproducibility:** This shall be limited to a value no greater than  $10\ \mu\text{W}$ .
- g) **Temperature Measurement:** Temperature accuracy shall be equal to or within  $\pm 0.1^{\circ}\text{C}$ ; temperature precision shall be equal to or within  $\pm 0.1^{\circ}\text{C}$ .
- h) **Calorimetric Measurement:** Calorimetric precision shall be equal to or better than  $\pm 1\%$ , based on metal standards; calorimetric sensitivity shall be equal to or better than  $0.2\ \mu\text{W}$ . Peak width resolution shall be  $0.32^{\circ}\text{C}$  or better.
- i) **Sample press:** Crimping dies and press to hermetically seal DSC sample pans.

## 2.0 Requirements for the Thermogravimetric Analyzer (TGA)

- a) **Temperature Range:** Achievable temperature shall be from ambient (room) temperature (approximately  $25^{\circ}\text{C}$ ) to a temperature no less than  $950^{\circ}\text{C}$ .
- b) **Weighing Capacity:** A maximum sample mass no less than 1 gram.
- c) **Balance Sensitivity:** A value no greater than  $0.1\ \text{mg}$
- d) **Weighing Precision:** A value no greater than  $0.01\%$  at  $100\ \text{mg}$  sample mass.
- e) **Baseline Drift:** This shall be no greater than  $50\ \mu\text{g}$  from ambient to  $900^{\circ}\text{C}$ , without baseline subtraction.
- f) **Purge Gas Control:** This capability shall be available using digital mass flow control, and programmable using the same operating software that controls other operational features of the TGA, including the ability to save this information as a separate signal in the TGA data file. Automated gas switching during data acquisition must also be achievable. A short (under 3 cm) exhaust path shall be available in the unit, to enable off-gas analysis.
- g) **Cooling:** The capability to cool from  $900^{\circ}\text{C}$  to ambient temperature in less than 15 minutes shall be included.

## 3.0 ARES Upgrade: Modify existing Rheometrics ARES mechanical spectrometer to provide following capabilities:

- a) Increase data acquisition rate to at least 100 data points per second.
- b) Provide external digital input and output lines, with data acquisition time (synchronized) to test events (e.g., the initiation of a particular test protocol)

#### **4.0 Computer Software Specifications**

Data acquisition and analysis software shall, at minimum, provide the following:

- a. Software should be compatible with (user-supplied) Intel-based PC running Windows XP OS.
- b. Real-time data analysis
- c. Autoanalysis
- d. Capability of providing at least eight (8) simultaneous curve overlay plots,
- e. Capability of exporting data into Microsoft Word or Excel files, spreadsheet text files, and ASCII data files.

#### **5.0 Installation and Training**

- a) The price of the thermal analysis system must include delivery of the instrument to NRL, Washington, D.C., including shipping insurance for the full value of the contents; and installation at NRL, Washington, D.C. Installation shall include a demonstration that the instrument is in compliance with the specifications.
- b) At the completion of the installation and demonstration of the specifications, the successful offeror must provide on-location training at NRL for 4 people for a minimum of two full days to familiarize the operators with proper operation and care of the instrument. Any additional training offered should be clearly identified in the proposal.

#### **6.0 Documentation and Warranty**

- a. A full set of all written documentation customarily provided to the public with a commercial item shall be provided. This shall include one hard copy of all operations and maintenance manual(s) or equivalent as well as copies of any software, and any manuals for the software included with the system, if customarily provided. This documentation must be received at NRL with the system hardware, unless other arrangements are agreed to by the authorized Government representative.
- b. The contractor shall offer the Government at least the same warranty terms, including offers of extended warranties, offered to the general public in customary commercial practice. These warranty terms must be included in the system price. The period of the warranty shall begin upon acceptance.