Award Period: From 7/1/84 To 6/30/85

Type Agreement: Grant No. AFOSR-84-0183

Sponsor Amount: Estimated: $53,000  Funded: $53,000

Cost Sharing Amount: $27,775

Title: Combustion Dynamics of Solid Propellants

Administrative Data

1) Sponsor Technical Contact:
Leonard H. Cavenly
AFSOR/NA
Building 410
Bolling AFB, DC 20332

2) Sponsor Admin/Contractual Matters:
Valerie M. Spencer
AFOSR/PK
Building 410
Bolling AFB, DC 20332

Travel: Foreign travel must have prior approval — Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with GIT

Cost Sharing No: E-16-318

Comments:
*Includes $7,520 cost sharing from Perkin-Elmer Corp. (23.5% of costs of thermal analysis lab-$17,000, TGA unit-$15,000)
Date 11/4/86

Project No. E-16-667

Includes Subproject No.(s) N/A

Project Director(s) E. W. Price

Sponsor AFOSR Bolling AFB, DC 20332

Title Combustion Dynamics of Solid Propellants

Effective Completion Date: 6/30/86 (Performance) 8/30/86 (Reports)

Grant/Contract Closeout Actions Remaining:

- [ ] None
- [X] Final Invoice or Final Fiscal Report
- [ ] Closing Documents
- [X] Final Report of Inventions - Questionnaire sent to P.I.
- [X] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other

Continues Project No. Continued by Project No.

COPIES TO:

- Project Director
- Research Administrative Network
- Research Property Management
- Accounting
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- Research Security Services
- Reports Coordinator (OCA)
- Library
- GTRO
- Research Communications (2)
- Other I. Newton
- Project File
- R. Embry
- A. Jones
- Other
STATUS REPORT

ON DOD/AFOSR Grant AFOSR-84-0183, Amend. A.

9 December 1985

Edward W. Price
School of Aerospace Engineering
Georgia Institute of Technology
Atlanta, GA 30332
The School of Aerospace Engineering at Georgia Tech has been conducting research on combustion of solid rocket propellants since 1968, and has developed a substantial capacity in that area in terms of qualified faculty, experimental facilities, and ongoing contracted research. The commitment to this area has been strongly supported by ONR and AFOSR, as well as by Georgia Tech administration, and a continuing high level of activity is planned. In addition, the technical and experimental capability in the propellant combustion laboratory has potential for much broader application relevant to DoD and other federal agencies. As an example, a study was started this year for ARO on the combustion mechanisms of thermite systems. Other facilities in the School of Aerospace Engineering conduct research in all combustion areas, for DoD, DOE, EPA, ERI, GM, etc.

As part of the strategy for propellant combustion research, development has been undertaken of a facility for study of the decomposition of individual propellant ingredients under conditions controlled to simulate combustion, but without the complexity of the full combustion system. This strategy permits better control of experimental conditions, better measurement of sample response, and hence less equivocal interpretation of results. Facilities include an optical hot stage microscope, thermogravimetric analyzer and DTA, an in-house-developed high heating rate (100°C/sec) thermogravimetric analyzer, and a CO₂ laser facility for very high rate pyrolysis. The present DoD Equipment Grant was established to support development of this high temperature decomposition facility, with some lesser items applicable to the associated propellant combustion facilities. This report is on the status of proposed and actual equipment purchases on the Equipment Grant, AFOSR Grant # 84-0183.
Progress

Attachment A shows the original list of equipment requested in the proposal. Columns on the right indicate the status of action on purchase. Numerical entries indicate actual expenditures. Items with no numerical entry indicate that no purchase has been made. An asterisk indicates significant change in planned or actual expenditure, and such items are described below, where status of each item is described, and the rationale for any changes or inaction is described. This is followed by a section on Plans, which indicates proposed action on outstanding items, and describes briefly what is proposed in another DoD Equipment Proposal that was recently submitted to cover further development of this facility.

Item #1 -- CO₂ laser. A laser purchase has been made, and installation is in progress. At the urging of the grant technical monitor, a market search was made for a more powerful laser (- 1000 watt vs proposed 500 watt). Costs were much higher, but a decision was made to purchase a Penn Research Corporation fast axial flow 1300 watt laser. The list price for this laser is $122,500 as compared to the $74,000 for the originally proposed 500 watt laser. This is an industrial laser of modern design and operational flexibility that has seen enough industrial use to establish high reliability. The manufacturer has provided a $25,900 grant to Georgia Tech to help cover the cost. Thus, the final cost is $22,600 in excess of the originally budgeted cost. The technical monitor concurred on the decision to go to the more costly laser. The added cost is being met by dropping Item #5 in Attachment A (see below). Item #1 has also incurred appreciable installation costs (gas supply, electrical hook-up, cooling water hook-up, and heavy duty movers' services). The estimated cost of these items (not all billed yet) is approximately $6,000, putting costs at $28,600 over the budget amount for this part of Item #1.

The decision to obtain a larger laser necessitates reconsideration of the choice and cost of the optical train (Item #1 -- $9,000). A choice on optics has been delayed pending further design of the experimental set-up. A more ambitious design of the set-up has been proposed to capitalize on the potential of the laser and accommodate more users. This plan is for a working area with three work stations and added equipment for observation of test sample response to
laser heating. This set-up requires more laser optics than originally proposed, and this is the object of a current request for a second equipment grant. It is proposed that the previously budgeted funds for an optical train be used to equip the first work station for exploratory work on ongoing contracts (ONR and ARO, possibly SDI), with optics suited to the larger CO₂ laser.

Item #2 -- Optical Pyrometer. Action on this item has been delayed pending determination of the final cost for the CO₂ laser plus installation. The added costs of Items 1 and 3 have reduced the funds available for this item.

Item #3 -- Thermal Analysis Lab. Purchase of this system was delayed due to changes in the supplier's list price and reluctance of the supplier to provide the promised donation toward purchase. The original proposal for the grant showed an estimated cost of $32,000 and a supplier's grant of $7,250 (net cost $24,750). The figures finally negotiated were: cost, $38,628; supplier's grant, $9,283; net cost, $29,395. This apparatus has been purchased and is in routine use. This apparatus provides the conventional low heating rate decomposition data typical of most published sources, and will be used as a basis for comparison for data to be obtained from the two higher heating rate experiments under development with the support of the present grant.

Item #4 -- Semi-Micro Balance. Purchased and in service.

Item #5 -- Surface Profile Analyzer. Purchase was held up because further evaluation showed the instrument would not provide the measurements of test surfaces needed on this project. A search was made for a suitable instrument without success. It is proposed that funding for this item be applied to Item #1 to meet the added cost of the larger CO₂ laser.

Item #6 -- Particle Segregator. Purchased and in service.

Item #7 -- Microscope Mirror Housing. Purchased and in use.

Item #8 -- Analytical Balance. Purchased and in use.

Item #9 -- Projector (16 mm motion analyzer). Purchased and in use.
The objective of the High Temperature Decomposition Facility was initially to support ongoing contracts on propellant and thermite combustion. The strategy was to be able to conduct pyrolysis and ignition tests under a wide range of sample heating rates from conventional TGA and DTA (max temperature rise rate - 1°C/sec) to the high heating rates in propellant combustion (10^5 0°C/sec). The Perkin-Elmer Thermal Analysis Lab (Item 3) has accommodated the low heating rate end of the desired range and provides the point of comparison of results from the higher heating rate experiments with the body of published literature. A second system has been under development on an ONR contract that will provide time resolved sample mass-time measurements for tests at heating rates up to 10^2 0°C/sec. Atmospheric pressure tests with this unit indicate that it will be ready for routine use around February 1986. The CO2 laser is the third system, and can be used as a heat source for many experiments. The immediate objective is to be able to expose the end of a 5 mm rod of polymer to uniform flux sufficient to produce a surface regression rate of 0.5 cm/sec. This is a typical rate for the binder in a composite propellant, and corresponds to a surface temperature rise rate of 10^5 0°C/sec near the sample surface. Surface recession rate and surface temperature would be measured over a range of heating rates, and the effective decomposition kinetic constants would be determined.

The effort to achieve decomposition data at high temperatures (which requires tests at high heating rate) is motivated by evidence that decomposition behavior is different at combustion zone temperatures than in conventional decomposition experiments. There is a desperate need for high temperature data in all combustion problems, and in many other problems where combustion is a secondary issue or completely absent. Thus the facilities under development will permit research on combustion, hazards, powder sintering, material degradation at high temperature, etc. Studies were recently started (for ARO) on the mechanism of propagation of reaction waves in powder mixtures of metals and metal oxides. The CO2 laser facility will be particularly useful in that work because it will be possible to produce states and processes normally present in the combustion wave, in non-burning samples, and then carry out microscopic studies of the samples after abrupt interruption of heating. Lacking such a method, there has been no way to gain any direct information about how the powder particles interact in the combustion zone of these systems, leaving the state of the theory on very speculative grounds.
The present plan is to have the CO₂ laser operational on rudimentary experiments by about 1 March 1986, in support of both ONR (combustion of propellants) and ARO (combustion of metal-metal oxide powders) contracts, and possibly on a proposed SDI contract on combustion efficiency of metals in solid rockets. The part of Item #1 that is uncommitted to date (laser beam delivery system) will be required for laser optics for this work. A request has been made for a second DoD equipment grant, which would provide for multiple test stations on the laser facility and components for test setups. This request also seeks funding for an infrared spectrometer, to be used for time-resolved composition analysis of gases from test samples in all of the pyrolysis experiments (especially the laser heated experiments).

It is proposed that the other principal item of the proposal that has not been purchased (Item 5) be cancelled (see previous explanation), and that the funds be allocated to the higher cost of the more powerful CO₂ laser (we have consulted with the technical monitor and proceeded on this basis).
### Equipment Budget

**(High Temperature Decomposition Facility)**

<table>
<thead>
<tr>
<th>Item</th>
<th>Source</th>
<th>Equipment</th>
<th>Cost (proposal estimate allowing for supplier's contribution)</th>
<th>Final Supplier's List Price</th>
<th>Cost to GIT</th>
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<tbody>
<tr>
<td><strong>RAPID HEATING EQUIPMENT</strong></td>
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<tr>
<td>1.</td>
<td>Photon Sources, Inc. 12165 Globe Road Livonia, MI 48150</td>
<td>Laser, C.W. CO Model 8500-2 Optical Train Installation</td>
<td>74,000. 122,500. 25,900. <strong>96,600.</strong></td>
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<td><strong>9,000.</strong></td>
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<td><strong>THERMAL ANALYSIS EQUIPMENT</strong></td>
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<td>2.</td>
<td>Barns Engineering, Inc. 30 Commerce Road Stamford, Conn. 06904</td>
<td>Optical Pyrometer Model RM-2A 1500 C Extender 1000 degree Black Body Variable Chopper</td>
<td>19,300.</td>
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<tr>
<td><strong>CHARACTERIZATION EQUIPMENT</strong></td>
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<tr>
<td>3.</td>
<td>Perkin-Elmer Corp. 510 Guthridge Court Norcross, GA 30092</td>
<td>Thermal Analysis Lab DSC-4 + System 4 TGA Unit</td>
<td>24,750. 38,628. 9,283. <strong>29,395.</strong></td>
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<tr>
<td><strong>GENERAL SUPPORT EQUIPMENT</strong></td>
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<tr>
<td>5.</td>
<td>ISI, Inc. 3255-6C Scott Blvd. Santa Clara, CA 95051 Bob Buchanan 800-538-6850</td>
<td>Surface Profile Analyzer Optical Comparator</td>
<td>25,000.</td>
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<tr>
<td>6.</td>
<td>Gibson, Inc. P.O. Box 877 Worthington, OH 43085 Doug Armstrong 614-846-5979</td>
<td>Particle Segregator ATM Sonic Sifter AT-3 Horz. Pulse Unit Steve #170 Steve #325 Steve 30um-50um</td>
<td>6,975. 7,604.</td>
<td><strong>---.</strong></td>
<td><strong>7,604.</strong></td>
</tr>
<tr>
<td>7.</td>
<td>Washaw Scientific, Inc. 5500 Oakbrook Pky Norcross, GA 30093 Tim Marshall 404-447-5632</td>
<td>Microscope Mirror Housing Leitz 514-391</td>
<td>1,800. 1,800.</td>
<td><strong>---.</strong></td>
<td><strong>1,800.</strong></td>
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<tr>
<td>8.</td>
<td>Fisher Scientific, Inc. Ibid.</td>
<td>Analytical Balance Mettler AK-160</td>
<td>6,000. 2,033.</td>
<td><strong>---.</strong></td>
<td><strong>2,033.</strong></td>
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</table>

**Note:** Matching funds are based on:
- Georgia Tech Research Institute (GTRI) 25% of Total Cost.
- Perkin-Elmer, Inc., 23.5% of Cost of Item 3.

* See discussion in text.

**After supplier contribution.
Combustion Dynamics of Solid Propellants (Uncl.)

E. W. Price

Scientific & Final

FROM 7-1-84 TO 6-30-86

1986, October 15

Development of facilities for studies of high temperature decomposition of solid propellant ingredients are described, particularly as they pertain to use of the subject DoD Equipment Grant. Funding from the Grant was used for laboratory support equipment and peripheral equipment on existing experiments, but primarily for purchase of a 1200 watt CO₂ laser and related beam optics and support equipment. This apparatus will permit heating of ingredient samples at surface heat fluxes common in propellant combustion, involving temperature rise rates of 10⁸ °C/sec at the surface. Combustion research indicates that decomposition mechanisms are usually different in such high heating rate situations, giving considerable urgency to development of high rate experiments. Several other planned uses for the laser facility are also noted.
SCIENTIFIC AND FINAL REPORT

COMBUSTION DYNAMICS OF SOLID PROPELLANTS

By

E. W. Price
Georgia Institute of Technology
School of Aerospace Engineering
Atlanta, Georgia 30332

Prepared for

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH
BOLLING AIR FORCE BASE, DC 20332

Under Contract DoD/AFOSR GRANT AFOSR-84-0183, AMEND. A

October 1986
BACKGROUND

Georgia Tech has maintained research studies in the School of Aerospace Engineering on combustion of rocket propellants and energetic systems since 1967. Contracted research has been sponsored by Navy, Air Force, Army, NASA, and industrial funding as well as University funding. A laboratory in the School of Aerospace Engineering has been equipped for this research, and approximately 35 graduate degrees have been awarded to students conducting research in this area. This specialized research is carried out in the context of a broader research and teaching program in aerospace propulsion and related scientific disciplines involving some 10 teaching faculty and 12 engineering staff members.

Combustion of solid propellants and solid ramjet fuels (and filled polymers in general) involves decomposition of the solid ingredients to vapor products, which react exothermally in the gas phase to provide all or part of the heat that sustains the surface pyrolysis. The pyrolysis rates of the solids that are required for practical applications require temperatures around 600-700°C. In the combustion environment, these temperatures are reached very quickly in a thermal wave that propagates into the solid ahead of the "burning" surface. Only in the presence of such rapid heating (e.g., $10^5$ °C/sec) is it possible to have decomposition at such high temperatures. At lower heating rates, the sample is gone before such temperatures are reached.

The propellant combustion wave is a very difficult environment in which to study ingredient decomposition, so many experiments have been contrived that are more suitable for conduct of measurements and analytical description of processes. Such methods include self-deflagration of ingredients that can sustain such behavior (e.g., ammonium perchlorate), combustion of geometrically or chemically simplified systems (e.g., "sandwich" burning and fuel combustion in oxidizing gas environments), and decomposition during externally controlled heating. All of these methods are in use together in the studies of propellant combustion at Georgia Tech. Each method has its own merits and weaknesses, but collectively they offer the prospect of definitive understanding of propellant combustion. The merit of the simpler experiments derives from their susceptibility to more definitive control of variables, measurement of processes, and interpretation of results. However, in the present context, the value of results depends on their relevance to propellant combustion. Relevance can easily be unintentionally sacrificed in the pursuit of "measurability" and
understandability". It is increasingly evident that this has happened in the area of controlled decomposition experiments, and the central objective of the facility development under the present contract has been to provide ingredient decomposition experiments that provide testing conditions that better simulate the combustion zone environment. The primary thrust to date is to develop experimental facilities that will permit rapid controlled heating of test samples to temperatures in the 500-1000°C range, and to measure the rates of decomposition and the composition of decomposition products in the absence of complicating combustion reactions. Current plans call for experiments in inert gas at atmospheric pressure. Later, work will be extended to lower and higher pressure. These goals were set as a result of findings in past and current DoD contracts (Refs. 1-4), and led to the proposal on which the present contract was based. In the following, a description will be made of experimental methods in use in the ongoing research, and the use of the DoD Equipment Funds to equip test facilities for that research. Some other potential applications of the facilities will also be listed to suggest lines of future research. A subsequent section of the report will describe what was purchased under the present contract, and the present and projected utilization.

DECOMPOSITION EXPERIMENTS

The objective of any high temperature decomposition experiment is to produce a controlled heating of a sample in a chosen environment, and to observe what happens to the sample. The experiments can be made relatively simple by "suitable" choice of the heating and environment, and by limiting the tests to simple materials. For example, if the sample is heated slowly enough, it will be easy to measure its temperature, and the temperature will be spatially uniform so that the temperature time history of all of the sample will be the same. Temperatures will be limited to relatively low values that can be reached before the sample is vaporized. When high temperature decomposition is sought, samples must be heated rapidly. It is not generally possible to maintain spatially uniform temperatures in a sample when it is heated rapidly, and it is difficult to determine the space-time-temperature history. The present facilities have struck three levels of compromise in choice of facilities:
1. **Conventional experiments** in which samples are heated by electrical heaters at rates of up to $1^0\ C/\text{sec}$ (hot stage microscope (optical and scanning electron types), differential thermal analyzer, thermogravimetric analyzer).

2. A relatively high heating rate thermogravimetric analyzer (HHRTGA), designed and built in this lab (Ref. 2), that can use radio frequency inductive heating or laser heating and make time-continuous mass and temperature measurements for heating rates to $10^2\ C/\text{sec}$ (spatially uniform sample temperature is achieved by using very thin samples).

3. A very high heating rate decomposition facility that utilizes a 1200 watt $\text{CO}_2$ laser to produce heating rates to $10^5\ C/\text{sec}$ (higher rates with a focused beam). By this means, test samples will be heated by passage of a thermal wave similar to that during combustion, and pyrolysis rate will be measured by surface regression rate and/or sample weight change. The heating history of individual volume elements will be determined from measured surface temperature and surface regression rate combined with calculation of corresponding temperature-time histories (which will be verified by thermocouples embedded in the samples).

The three types of facilities noted above are useful for developing different kinds of information about how samples decompose. The conventional systems are relatively easy to operate, and useful for determination of properties of samples such as temperatures of crystal phase change and/or melting, onset of vaporization, and estimates of energies of phase changes and apparent activation energies of decomposition reactions. Since most published literature is based on these low rate experiments, it was deemed advisable to upgrade the capability in this laboratory so that results of high rate tests could be compared with the "conventional data base" (to determine the conditions under which high heating rates and temperatures lead to different decomposition mechanisms). The upgrading included purchase of a Perkin and Elmer Thermal Analysis Lab Model 7/4; purchase of accessories for an available Leitz optical microscope and its camera attachments for use as a hot stage microscope. A video recorder was purchased for use with the available scanning electron microscope, to record sample behavior during heating (in vacuum).
The high heating rate thermoanalyzer was developed with ONR Contracts N00014-79-C-0764 and N00014-85-K-0803 and is described in detail in Refs. 1 and 2. The test sample is deposited as a thin film on a ferroelectric sample holder that is heated by RF induction. Temperature is measured by a thermocouple attached to the sample holder. RF energy is provided by a Fischer 0310 Curie Point pyrolizer unit (The sample was also heated with the heater unit from the Perkin and Elmer system for comparison). The heater element is mounted on the free end of a vibrating quartz tube, and the change in sample mass is measured by the change in frequency of the vibration. The system was used to determine the effective activation energy of HTPB and PBAN binders at high and low heating rates. The indicated activation energy of HTPB was \( \sim 80,000 \) cal/mole at a heating rate of 100\(^\circ\) C/sec, as compared to a value of \( \sim 18,000 \) at conventional heating rates, e.g., 1\(^\circ\) C/sec. PBAN binder showed the same low value of activation energies as HTPB, but at both heating rates. This result indicates the importance of obtaining decomposition behavior at high heating rate. The low activation energies appear to reflect rate control by evaporation, while the high activation energy appears to reflect rate control by chemical bond breaking. Under the present contract, the primary support of this HHRTGA facility was the purchase of three amplifiers for use in the data acquisition systems.

The CO\(_2\) laser pyrolysis facility is intended for a variety of ignition, pyrolysis, and combustion experiments (Ref. 5). The immediate applications are to ONR Contract N00014-85-K-0803 (ingredient pyrolysis) and ARO Contract DAAG 29-85-K-0125 (thermite combustion). This facility is still under construction; the laser is operational and in use with improvised experimental setups, while the optical train, control apparatus, test chambers, and measurement systems are under construction. This facility has been the primary cost item under the present Equipment Grant. A decision was made in concert with the AFOSR Technical Monitor to acquire a 1200 watt laser instead of the proposed 500 watt unit, a decision based on potential added usefulness and advances in available commercial lasers. The manufacturer also provided a $25,900 grant toward the purchase. This facility has already made it possible to obtain controlled ignition, laser-assisted burning, and interrupted burning of metal-metal oxide samples for the above-noted ARO contract. When completed, the facility will provide a uniform beam for the pyrolysis of polymer (fuel) samples at temperature rise rates of \( 10^5 \) \( ^\circ\)C/sec. A follow-on DoD equipment grant is concerned with instrumentation for measurement of sample behavior during the heating-pyrolysis
event (time resolved temperature and composition fields during high rate pyrolysis and oscillatory pyrolysis).
CURRENT AND PLANNED RESEARCH

The high temperature decomposition facility provides the means for a wide range of research, not only in propulsion applications but in all high temperature applications and problems from fire research to determination of material properties, to metallurgy, to material failure. The propulsion-related problems currently planned or in progress are:

1. Decomposition of polymeric materials, such as propellant binders and ramjet fuels (in progress),
2. Combustion of "thermite" materials (gasless combustion of metal-metal oxide and metal-metal systems) (in progress),
3. High temperature behavior of particulate burning rate catalysts, such as Fe₂O₃, and mechanisms of catalysis (in progress),
4. Sintering and agglomeration processes in metal powders used as fuels in propulsion,
5. Oscillatory combustion using oscillating laser beam as a controlled perturbation source,
6. Decomposition of solid oxidizers: topochemistry of crystal decomposition, presence of melts at high temperature, composition of vapor products,
7. Laser assisted combustion to permit combustion at low pressure with extended combustion zone (making space-resolved combustion zone diagnostics practical),
8. Ignition mechanisms, ignition energy requirements.
EQUIPMENT PURCHASED AND INSTALLED

A listing is provided below of equipment purchases and installation costs charged to this Grant and Georgia Tech matching funds. The items are grouped according to the particular experimental facilities noted in the text.

**CO₂ Laser Pyrolysis Facility**

- Penn Research Corporation, 1200 Watt Laser 122,500
- Laser Beam Benders and Lens 12,661
- Optical Bench 5,314
- Gas Regulators 1,105
- Exhaust System Blower 1,357
- Water Chiller 2,500
- Installation costs (moving, electrical, water hook up) 1,132

**Thermal Analysis Lab**

- Perkin and Elmer DTA, TGA (System 7/4) 29,395

**Optical Hot Stage Microscope**

- Leitz Mirror Housing 1,760
- Motor Drive and Film Magazine for Nikon Camera 1,137
- Lens for Nikon Camera 170
- Lens for Lo Cam Camera 150

**Scanning Electron Hot Stage Microscope**

- Video Recorder 300

**High Heating Rate Thermogravimetric Analyzer**

- 3 Neff Amplifiers 2,471
### Test Sample Preparation Equipment

<table>
<thead>
<tr>
<th>Equipment</th>
<th>Quantity</th>
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<tbody>
<tr>
<td>Mettler Balance</td>
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<td>Milli Balance (Cahn DTL 7500)</td>
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<td>Sonic Sifter</td>
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<td>Micro Sieves</td>
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<td>High Temperature Bath</td>
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### General Lab Equipment

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<th>Equipment</th>
<th>Quantity</th>
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<tr>
<td>Lockable Storage Cabinets (five)</td>
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<tr>
<td>Motion Picture Analyzer (16 mm stop motion projector)</td>
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<tr>
<td>Monochromatic Light (sodium)</td>
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$197,921$
REFERENCES


