



Users' Guide

PUB-475 c.2
August 1984

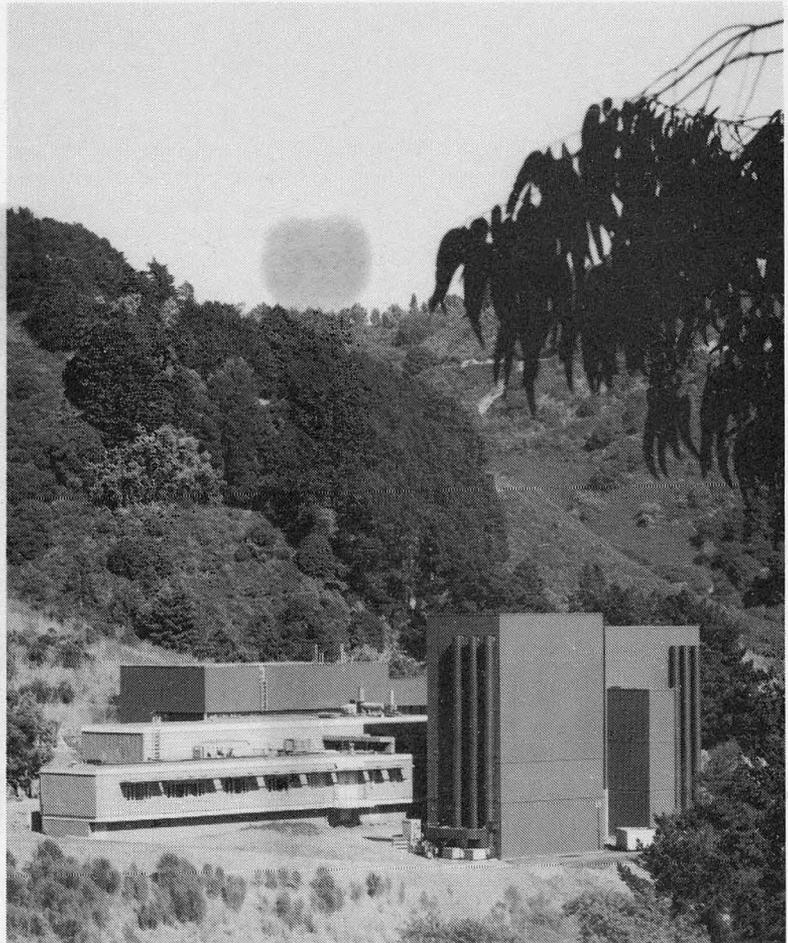
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Prepared for the U.S. Department of Energy under Contract No. DE-AC03-76SF00098



NATIONAL CENTER FOR ELECTRON MICROSCOPY
Lawrence Berkeley Laboratory
University of California

PUB-475
c.2

DISCLAIMER

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The National Center for Electron Microscopy (NCEM) in the Materials and Molecular Research Division of the Lawrence Berkeley Laboratory is a high voltage electron microscope facility for ultra high resolution or dynamic in-situ studies. It is supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Science Division, U. S. Department of Energy under Contract No. DE-AC03-76SF00098.

1. THE INSTRUMENTS

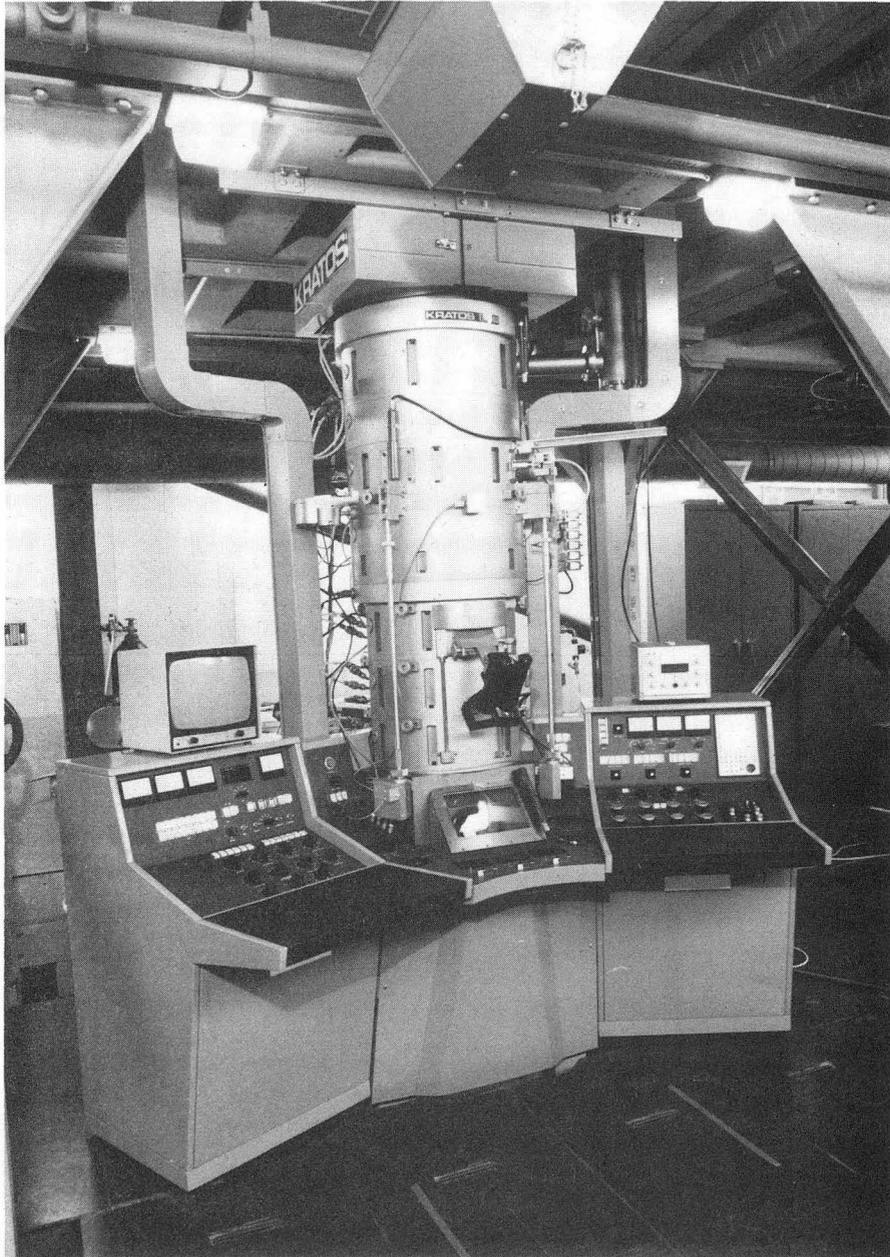
1.1. Kratos EM-1500

This instrument is designed primarily for dynamic in-situ studies but it can also resolve structures at the 3Å level. It is the first electron microscope to be built by Kratos with a maximum acceleration voltage of 1.5 MeV. As such, it provides accelerating voltages in the range 1200-1500kV not available elsewhere in the U.S. To achieve the higher accelerating voltage the number of stages of the Cockcroft-Walton generator was increased to 9 and the SF₆ insulating gas pressure was increased to ~90 p.s.i. Conditioning of the accelerator tube is carried out at 1.6 MeV. A series of 15 push buttons allows the accelerating voltage to be set at 150kV or 200-1500kV in 100kV increments. A photograph of the microscope operating level is shown in Fig. 1.

The more important instrument specifications are listed in Table I.

TABLE I: EM-1500 SPECIFICATIONS

Max ^m Accel. Voltage	1500
High Voltage Stability	$> 5 \times 10^{-6}/\text{min}$
High Voltage Ripple	$< 3 \times 10^{-6}$
Accelerator Vacuum	10^{-7}
Obj. Lens Current Stab.	$3 \times 10^{-6}/3 \text{ min}$
Focal Length	6.8mm
Spher. Abb. Coeff. (Cs)	10.5mm
Chrom. Abb. Coeff. (Cc)	5.4mm
NI/E ^{1/2} (at 1000kV)	11
Goniometer Tilts	$\pm 45^\circ$
Specimen Drift Rate	$< 0.3 \text{ \AA}/\text{sec}$ (0.5 hr.)
Vacuum	$\approx 10^{-7}$
└ Column	
└ Spec. Chamb.	$< 3 \times 10^{-6}$
External Vibr. (pk-pk)	$< 1 \mu\text{m}$
Resolution (pt-pt)	$\sim 3 \text{ \AA}$



CBB 807-8681

Fig. 1

The microscope is equipped with both a top-entry stage for high resolution studies, and a side-entry stage for general, dynamic in-situ, and environmental cell studies. Several different specimen stage rods are available (see Table II) and this inventory will be added to as demand develops and funding permits.

TABLE II: SPECIMEN STAGES FOR EM 1500

TYPE	X-TILT	Y-TILT	TEMPERATURE
<u>EM 1500</u>			
Double-tilt, cartridge-type	+45°	+45°	Ambient
Single-tilt, cartridge-type*		+45°	Ambient
Double-tilt, furnace type	+30°	+30°	Ambient-850
Single-tilt, furnace-type*		+25°	Ambient-850

* For use with Environmental Cell at pressures up to 1 atmosphere

1.2 JEOL JEM-ARM-1000

This instrument has been specifically designed for ultimate performance in the high resolution imaging mode. Attention has been given to the potential problem of specimen sensitivity to the electron beam at higher accelerating voltages however, and by incorporating a microprocessor-controlled height (Z)-variable goniometer, the microscope will operate with a constant C_s product over its entire voltage range (400kV to 1000kV). Specifications for the Atomic Resolution Microscope (ARM) at maximum voltage are given in Table III; the instrument is shown in Fig. 2.

It is also important to note that at higher lens excitation values, the microscope can be run in condenser-objective mode, enabling convergent beam electron diffraction (CBED) patterns to be taken from the same specimen areas imaged in high resolution. For increased stability, the stage configuration on this machine is top entry, employing standard 3mm diameter specimen cartridges, and coupled to a microprocessor for precise stage manipulation. The microscope uses LaB_6 filaments for additional brightness and the camera accepts 50 film cassettes.

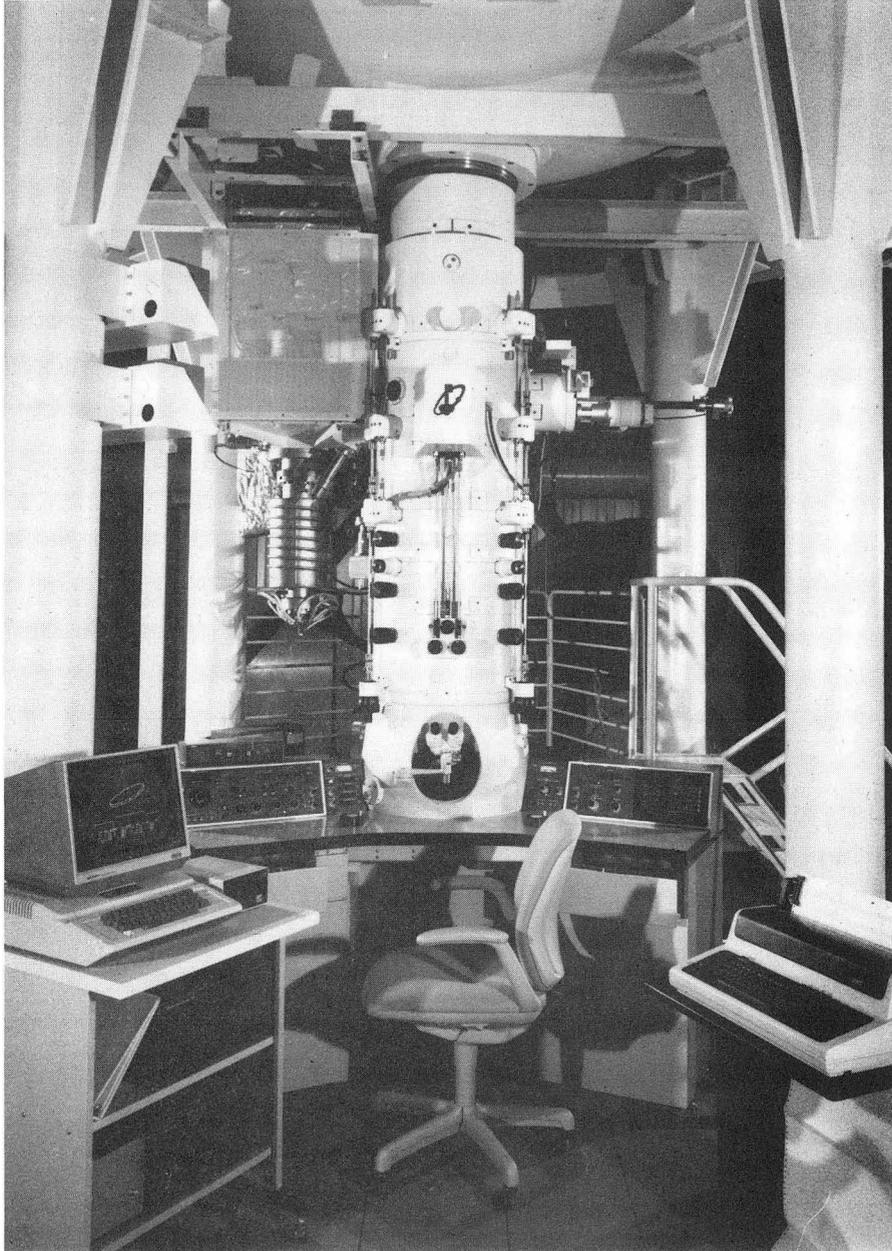


Fig. 2

CBB 839-8469

Table III: JEM ARM 1000 SPECIFICATIONS

Max ^m Accel. Voltage	1000kV
High Voltage Stability	$1 \times 10^{-6}/\text{min}$
High Voltage Ripple	$1 \times 10^{-6}/\text{min}$
Accelerator Vacuum	2×10^{-8}
Obj. Lens Current Stab.	$1 \times 10^{-6}/\text{min}$
Focal Length	2.6mm
Spher. Abb. Coeff. (Cs)	2.8mm
Chrom. Abb. Coeff. (Cc)	3.3mm
NI/E ^{1/2} (at 1000kV)	23
Goniometer tilts	$\pm 40^\circ$ biaxial with lift
Specimen Drift Rate	$.02 \overset{\circ}{\text{Å}}/\text{sec}$
Vacuum	Column
	Spec. Chamb.
External Vibr. (pk-pk)	$< 1 \mu\text{m}$
Resolution (pt-pt)	$< 1.7 \overset{\circ}{\text{Å}}$

1.3 JEOL JEM 200-CX

A support instrument to the ARM, the JEM 200-CX is well known for its high resolution performance. Relevant specifications for this machine, equipped with ultrahigh resolution goniometer (top entry), are given in Table IV. Note that specimens for this microscope must be 2.3mm in diameter in order to fit into the ultrahigh resolution pole piece.

Table IV. JEM 200-CX SPECIFICATIONS

Max ^m Accel. Voltage	200kV
Focal Length	2mm
Spher. Abb. Coeff. (Cs)	1.1mm
Chrom. Abb. Coeff. (Cc)	1.4mm
Goniometer tilts	$\pm 10^\circ$
Specimen Drift Rate	$< 0.5 \overset{\circ}{\text{Å}}/\text{sec}$
Vacuum	Column
	Spec. Chamb.
External Vibr. (pk-pk)	$< 3 \mu\text{m}$
Resolution (pt-pt)	$\sim 2.4 \overset{\circ}{\text{Å}}$

1.4 Hitachi HU-650 (see Fig. 3)

The accelerating voltage on this instrument is continuously variable between 200 and 650kV, but it is calibrated for, and most conveniently used at three fixed voltages, 300, 500 or 650kV.

The resolution limit of this instrument is not high (see Table V) but it is adequate for some studies, exploratory experiments, and many hot stage or environmental cell experiments (see Table VI).

TABLE V: HU-650 SPECIFICATIONS

Max ^m Accel. Voltage	650
High Voltage Stability	$10^{-5}/\text{min}$
High Voltage Ripple	$< 7 \times 10^{-6}$
Accelerator Vacuum	10^{-6}
Obj. Lens Current Stab.	$5 \times 10^{-5}/\text{min}$
Focal Length	8.4mm
NI/E ^{1/2} (at 1000kV)	12.8
Goniometer Tilts	$\pm 45^\circ$
Vacuum	Column 6×10^{-6}
	Spec. Chamb. $\sim 10^{-5}$
Resolution (pt-pt)	$\sim 20\text{\AA}$

TABLE VI: SPECIMEN STAGES FOR HU-650

<u>HU-650</u>			
Double-tilt, cartridge-type	$\pm 45^\circ$	$\pm 45^\circ$	Ambient
Single-tilt, cartridge-type*		$\pm 45^\circ$	Ambient
Double-tilt, furnace-type	$\pm 30^\circ$	$\pm 30^\circ$	Ambient-850
Single-tilt, furnace-type*		$\pm 25^\circ$	Ambient-850
Single-tilt, ribbon-type*		$\pm 30^\circ$	Ambient-1000
Single-tilt, straining*		$\pm 30^\circ$	Ambient

* For use with Environmental Cell at pressures up to ~ 300 torr

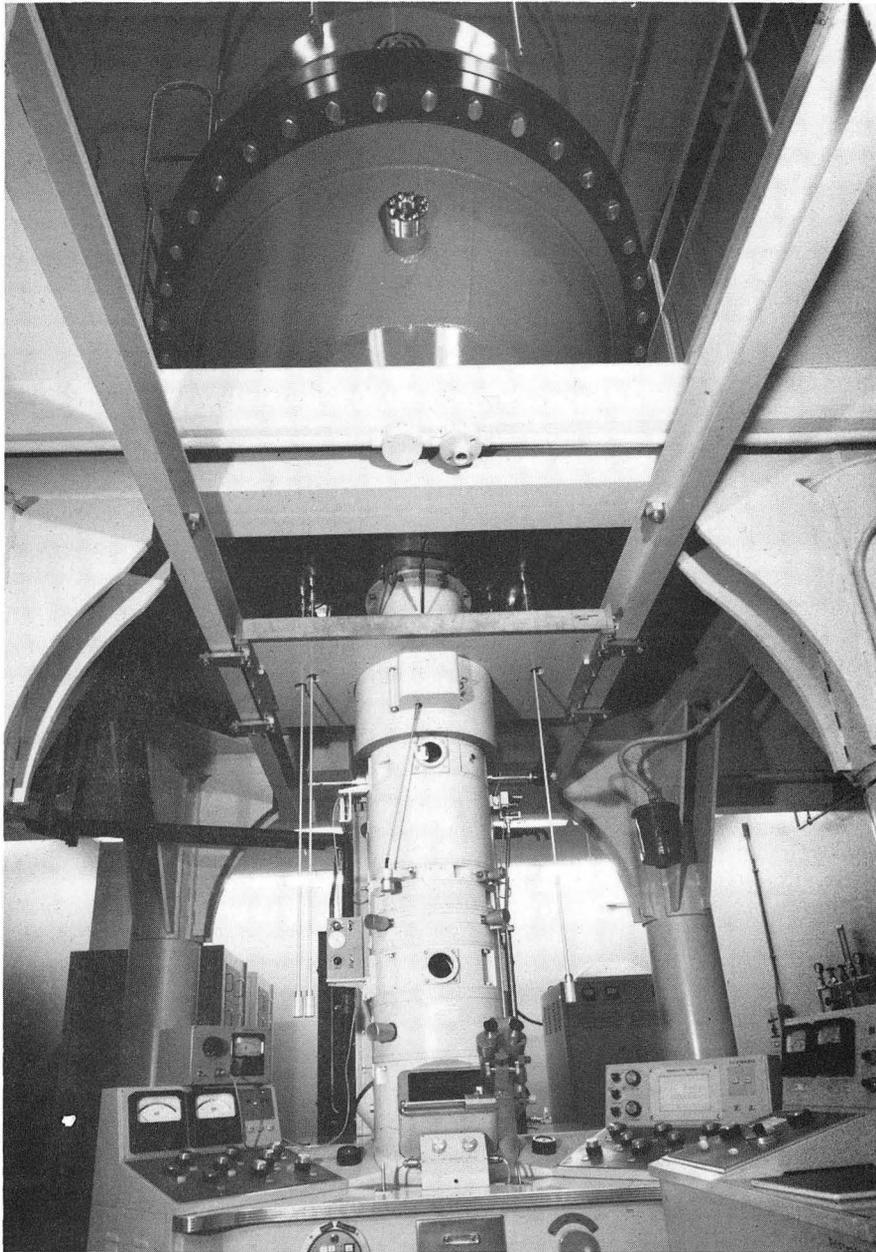


Fig. 3

CBB 701-360

2. USER POLICIES

Beam time on the HVEMs is allocated after review and acceptance of research proposals submitted in advance. The Steering Committee, composed of recognized scientists drawn from universities, industry and the national laboratories, meets at regular intervals to review, discuss, and accept project proposals and advise LBL management on Facility policies and operating procedures. The criteria used in assessing research proposals and assigning priorities are: appropriateness, scientific merit, and time availability. Proposals will remain valid for a period not to exceed 18 months. Scheduling beam time is the responsibility of the Facility Managers.

In accordance with established DOE policy, outside users must execute a Laboratory Guest Patent Agreement, an example of which is given in Appendix I.

No charges will be assessed for use of the HVEM Facility provided the proposed work is of documented programmatic interest to DOE and the results are published in the open literature. Items provided at no cost include time on the instrument, some staff assistance and advice, and use of dark rooms. Costs associated with use of effort and services outside the facility, e.g. machine shop, electronic shops, computer, stores, etc., must be borne by the user at the standard Laboratory internal rates.

If the proposed research is not of interest to DOE or is proprietary, full cost recovery (including overhead and DOE added factor) will be charged.

At the conclusion of an experimental program, regardless of the outcome, a brief report of the results and conclusions must be submitted to the Facility Managers. Copies of any publications resulting from the research must also be sent. These papers can be used to support applications for continuing or new experiments. They will also be useful as a permanent record of HVEM research activity.

Users must be qualified and experienced electron microscopists. It will be the responsibility of the Principal Investigator on the proposal to vouch for the competence of members of his team. Instructions sessions on the specific uses of the instrument will, of course, be given.

Any malfunctioning of the instruments must be reported (immediately, if serious in nature) to the Facility Support Staff or Managers. Unscheduled down-time and repairs will be minimized if all users operate the microscopes with care and consideration. Because of the disruption and inconvenience to other users, any damage to the instrument or stages resulting from operator ignorance or negligence will be repaired at the cost of the group responsible. If in doubt, seek assistance from the Facility Staff.

At least 24 hours notice is required if a microscope booking is cancelled. Failure to do so will result in loss of priority in subsequent scheduling.

3. PROPOSAL SUBMISSION AND SCHEDULING

A research proposal for using the ARM-1000 or EM-1500 should be accompanied by a Research Summary Form (RSF) (an example is shown in Appendix IV) and submitted at least 6 weeks prior to the earliest proposed starting date.

The proposal may be brief but should state clearly the scientific objective, and include use justification for the particular high voltage instrument, and a list of materials to be examined. Specimens should be prepared in advance. Any possible hazards associated with the specimens must be specified, and their possible effect on the microscope environment or specimen area must be evaluated, e.g., whether contaminating, radiation sensitive, volatile, likely to react with hot stage, etc.. Any special scheduling arrangements should be indicated on the RSF.

In the case of the ARM-1000, the proposal should be accompanied by high resolution micrographs taken on any conventional high resolution instrument together with their complete analysis and interpretation. This information will be used in assessing the need for the higher resolution or flexibility available on the ARM, and will also serve to demonstrate the operational skills of the applicants.

Experimenters may schedule time on a microscope by completing a second Schedule of Use Form (Appendix V) and return it to the Facility Administrator along with the research proposal. Non-local area users are encouraged to plan well in advance to allow local users to be assigned remaining time slots. Requests to use the environmental cell or to perform non-materials science experiments will be grouped and scheduled periodically to avoid excessive breaking of the column vacuum.

Users travelling long distances are advised to phone (415) 486-5006 for a late status report before departing.

4. SAFETY

RADIATION PROTECTION

Women Employees

The National Council on Radiation Protection (NCRP) has recommended that the total occupational radiation dose to the fetuses of fertile women not exceed 0.5 rem. It is Laboratory policy that fertile women working in radiation areas where it is possible to receive 0.5 rem or greater be notified, as well as their supervisors, of this recommendation and the reasons for it. It is DOE policy, however, that no woman be denied a job or work assignment solely because of the recommendation.

As soon as an employee learns that she is pregnant, she is required to inform Medical Services of the expected date of childbirth (see RMP, Sec. 2.09, D, 3). A physician will discuss her work environment with her and will determine whether or not any changes in the environment should be made to ensure her good health and that of her baby. All new employees are given a copy of the booklet, "Pregnancy and Radiation".

5. ACCOMMODATION AND TRAVEL

The responsibility for making travel and accommodation arrangements will rest with the user. However, the Facility administrative staff will be glad to give advice and assistance in making accommodation reservations when appropriate. There is a shortage of accommodation in the Berkeley area and a marked seasonal variation, so advanced reservations are highly recommended.

Appendix II lists accommodations covering a range of prices; Appendix III contains information on transportation to and from the airport together with area maps.

**UNIVERSITY OF CALIFORNIA
LAWRENCE BERKELEY LABORATORY
PATENT AGREEMENT**

This agreement is made by me with The Regents of the University of California, a corporation, hereinafter called "University", in part consideration of my employment, and of wages and/or salary to be paid to me during any period of my employment, by University, and/or my utilization of University research facilities.

By execution of this agreement I understand that I am not waiving any rights to a percentage of royalty payments received by University, as set forth in the University Policy Regarding Patents, hereinafter called "Policy". I further understand that I may, with the approval of the University, request a waiver determination by the U.S. Government on my identified inventions as set forth in 41 CFR 9-9.109-6, where applicable.

I agree that every possible patentable device, process, plant, or product, hereinafter referred to as "invention", which I conceive, make (first actually reduce to practice), or develop while employed by University, or during the course of my utilization of any University research facilities, shall be examined by University to determine rights and equities therein in accordance with the Policy, and I shall promptly furnish University with complete information with respect to each.

In the event any such invention shall be deemed by University to be patentable, and University desires, pursuant to determination by University as to its rights and equities therein, to seek patent protection thereon, I shall execute any documents and do all things necessary, at University's expense, to assign to University all rights, title and interest therein and to assist University in securing patent protection thereon. The scope of this provision is limited by California Labor Code section 2870, to which notice is given below, to the extent said Labor Code provision is consistent with federal law. In the event I protest the University's determination regarding any rights or interests in an invention, I agree: (a) to proceed with any University requested assignment or assistance; (b) to give the University notice of that protest no later than the execution date of any of the above-described documents or assignment; and (c) to reimburse the University for all expenses and costs it encounters in its patent application attempts, if any such protest is subsequently sustained or agreed to.

I shall do all things necessary to enable University to perform its obligations to grantors of funds for research or contracting agencies as said obligations have been undertaken by University, including the University's obligations regarding patents and technical and scientific records under Contract W-7405-ENG-48. (Contract-48) with the U.S. Government. With reference to Contract-48 I agree to abide by and fully perform the terms of Article XV of said contract, excerpts of which are set forth on the reverse side of this agreement, as they may be amended from time to time, to the extent applicable to me, and further agree that the Government shall have prior right to determine title to all such inventions, and that I will report all such inventions to the Director, Lawrence Berkeley Laboratory (LBL), or his designee. To protect the patent interests of the University and the Government, I agree not to publish any information regarding scientific or technical developments made or conceived in the course of or under Contract-48 without prior approval obtained from the Director, LBL, or his designee for this purpose.

University may relinquish to me all or a part of its right to any such invention, if, in its judgment, the criteria set forth in the Policy have been met.

I agree to be bound hereunder for and during any periods of employment by University or for any period during which I conceive, make (first actually reduce to practice), or develop any invention during the course of my utilization of any University research facilities.

In signing this agreement I understand that the law, of which notification is given below, applies to me, but that I am still required to disclose all my inventions to the University.

NOTICE

This agreement does not apply to an invention which qualifies fully under the provisions of Labor Code section 2870 of the State of California, to the extent said Labor Code provision is consistent with federal law. Said Labor Code provision provides that:

Any provision in an employment agreement which provides that an employee shall assign or offer to assign any of his or her rights in an invention to his or her employer shall not apply to an invention for which no equipment, supplies, facility, or trade secret information of the employer was used and which was developed entirely on the employee's own time, and (a) which does not relate (1) to the business of the employer, or (2) to the employer's actual or demonstrably anticipated research or development, or (b) which does not result from any work performed by the employee for the employer. Any provision which purports to apply to such an invention is to that extent against the public policy of this state and is to that extent void and unenforceable.

In any suit or action arising under this law, the burden of proof shall be on the individual claiming the benefits of its provisions.

Employee/Guest Name: _____
(Please Print)

Employee/Guest Signature: _____ Date: _____

Witness Signature: _____ Date: _____

**Excerpts from Modification No. 23
Contract No. W-7405-ENG-48**

(b) *Allocation of principal rights.* (1) Assignment to the Government. The University agrees to assign the Government the entire right, title, and interest throughout the world in and to each Subject Invention, except to the extent that rights are retained by the University under paragraphs (b) (2) and (c) of this clause.

(e) *Invention identification disclosures and reports.* (1) The University shall establish and maintain active and effective procedures to ensure that Subject Inventions are promptly identified and timely disclosed. These procedures shall include the maintenance of laboratory notebooks or equivalent records and any other records that are reasonably necessary to document the conception and/or the first actual reduction to practice of Subject Inventions, and records which show that the procedures for identifying and disclosing the inventions are followed.

(f) *Publications.* It is recognized that during the course of the work under this contract, the University or its employees may from time to time desire to release or publish information regarding scientific or technical developments made or conceived in the course of or under this contract. In order that public disclosure of such information will not adversely affect the patent interests of DOE or the University, patent approval for release or publication shall be secured from Patent Counsel prior to any such release or publication.

NOTE

At LBL, patent related duties and functions have been delegated by the Director to the LBL Patent Group. Employees are therefore requested to contact the LBL Patent Group for processing of patent matters.

Revised: December 1, 1980

July 1984

SUGGESTED HOTELS AND MOTELS

	<u>Minimum Rate/Single</u>	<u>Distance to LBL</u>
Berkeley House Motor Hotel 920 University Avenue, Berkeley (415) 849-1121	\$47.00 + tax	about 2 mi.
Durant Hotel 2600 Durant Avenue, Berkeley (415) 845-8981	\$48.00 + tax	adjacent to UC Campus
Marriott Hotel 200 Marina Blvd., Berkeley (800) 228-9290	\$78.00 + tax	about $2\frac{1}{2}$ mi.
Claremont Hotel 41 Tunnel Road, Berkeley (415) 843-3000	\$89.00 + tax	about 1 mi.
The Shattuck Hotel 2086 Allston Way at Shattuck Berkeley (415) 845-7300	\$39.00 + tax	about 1 mi.

OTHER TEMPORARY HOUSING

Men's Faculty Club* (415) 848-5678 - On UC Campus
 Single with private bath: \$45/day; \$38/day by the week; \$35 day by the month
 " " shared " 38 " 33 30
 (Use Lawrence Berkeley Laboratory as a "sponsor"--reserve a month in advance,
 if possible)

Women's Faculty Club* (415) 845-5084 - On UC Campus
 Single with private bath, private telephone and continental breakfast - \$40/night + \$1.25 weekly
 membership
 No weekly rates
 \$750/month (reserve a month in advance, if possible)

Bed and Breakfast Facilities--(415) 525-4569. This is a clearing-house for most of
 the B and B facilities in the area. We can specifically recommend Hank and Beta Lewis at
 853 Contra Costa Avenue.

SUMMER ONLY

International House (415) 642-9470 - UC Campus
 Mid-May until early August. Weekly rates only--\$196/week with meals
 (no rooms without meals)

University Guest Residence (415) 642-5925 after mid-May - Adjacent to UC campus;
 \$19.25/night single occupancy.

* These facilities are used by both women and men.

Lawrence Berkeley Laboratory is located in the hills east of the University of California, Berkeley Campus. Berkeley is served by both Oakland and San Francisco International Airports. More flights come into San Francisco (30 miles from LBL), but Oakland Airport is closer (15 miles away).

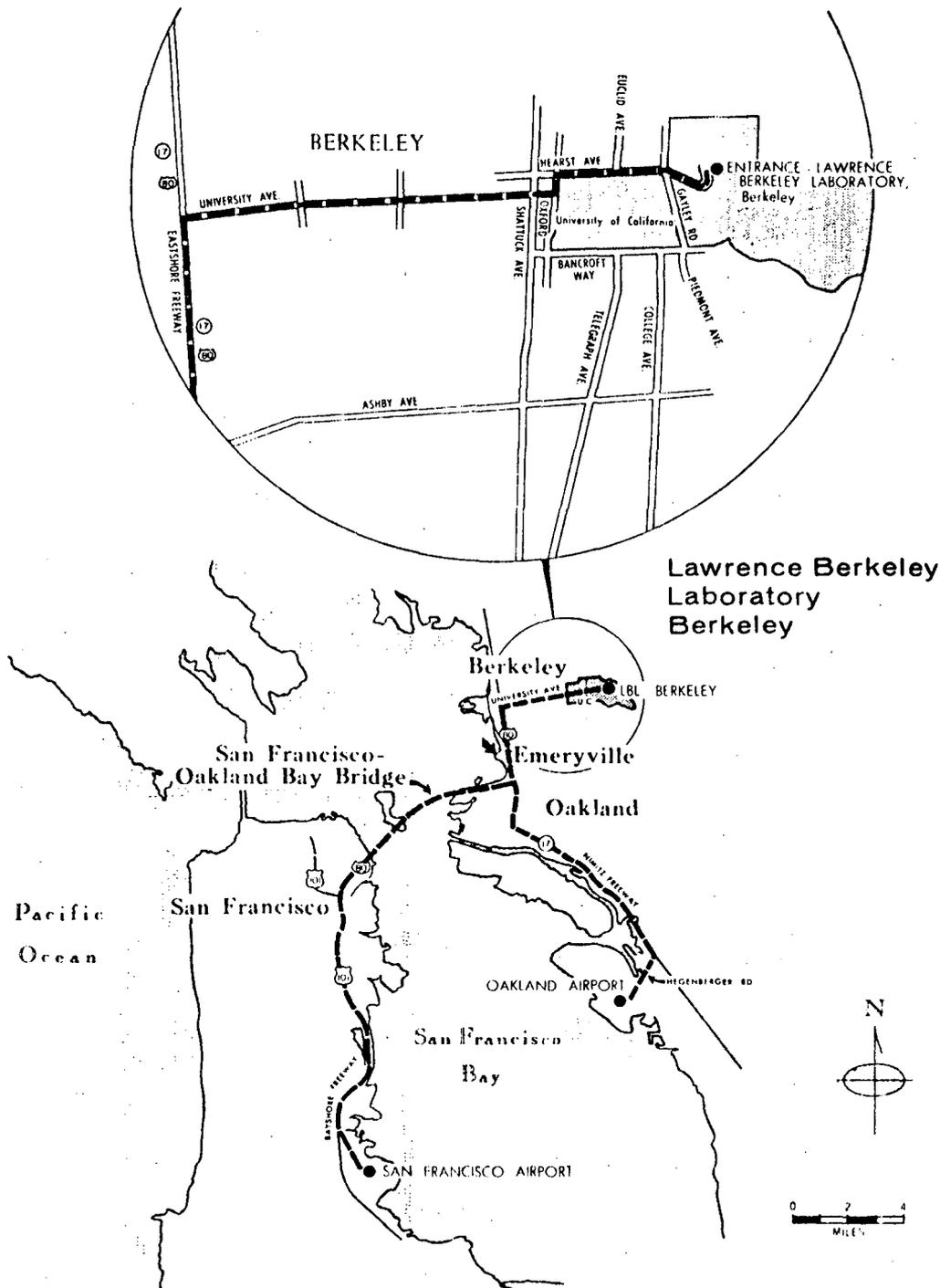
TRANSPORTATION FROM AIRPORTS TO BERKELEY

Airport Connection (415) 841-0150. Connects from San Francisco Airport to the Durant Hotel in Berkeley, next to the UC campus; the Marriott Hotel on the Berkeley Marina; Holiday Inn in Emeryville (just west of Berkeley), and the Claremont Hotel. \$12/person, 1-way. Reservations should be made in advance.

Taxi is about \$40.00 from San Francisco Airport; ~ \$22.00 from Oakland.

DIRECTIONS TO LBL STRAWBERRY CANYON GATE FROM BOTH AIRPORTS
(See attached map and directions)

1. The Facility (415-486-5006) should be notified previous to your arrival so that you will be allowed through the gate.
2. Guests should tell the guard they are going to Bldg. 72 to obtain credentials.



SAN FRANCISCO AIRPORT TO LBL-BERKELEY

Driving time: 45 min. to 1 hr.
Distance: about 25 miles

Upon leaving the airport, bear right and go north on Highway 101 to San Francisco-Oakland Bay Bridge approach (about 13 miles). Watch for signs for "Bay Bridge" and Interstate 80.

After crossing the bridge (about 4.5 miles), bear left and look for signs pointing to Interstate 80 and to Berkeley. (About 1.5 miles from the end of the bridge there is a major intersection from which Interstate 80 goes northward.)

Go north about 2.4 miles on Interstate 80 and take the University Avenue turnoff (right) into Berkeley. Stay on University Ave., about 2.6 miles, to its eastern end at Oxford St. (western side of U.C. campus), bearing left.

Turn left onto Oxford St. and, bearing right, take the first right turn onto Hearst Ave. and follow it up the hill to ~~LBL (Hearst Ave. becomes Cyclotron Road before the entrance to LBL).~~ *

From the east end of University Ave. to LBL is about 1 mile.

* Gayley Road. Turn right. See map.

OAKLAND AIRPORT TO LBL-BERKELEY

Driving time: 35 minutes
Distance: about 16.5 miles

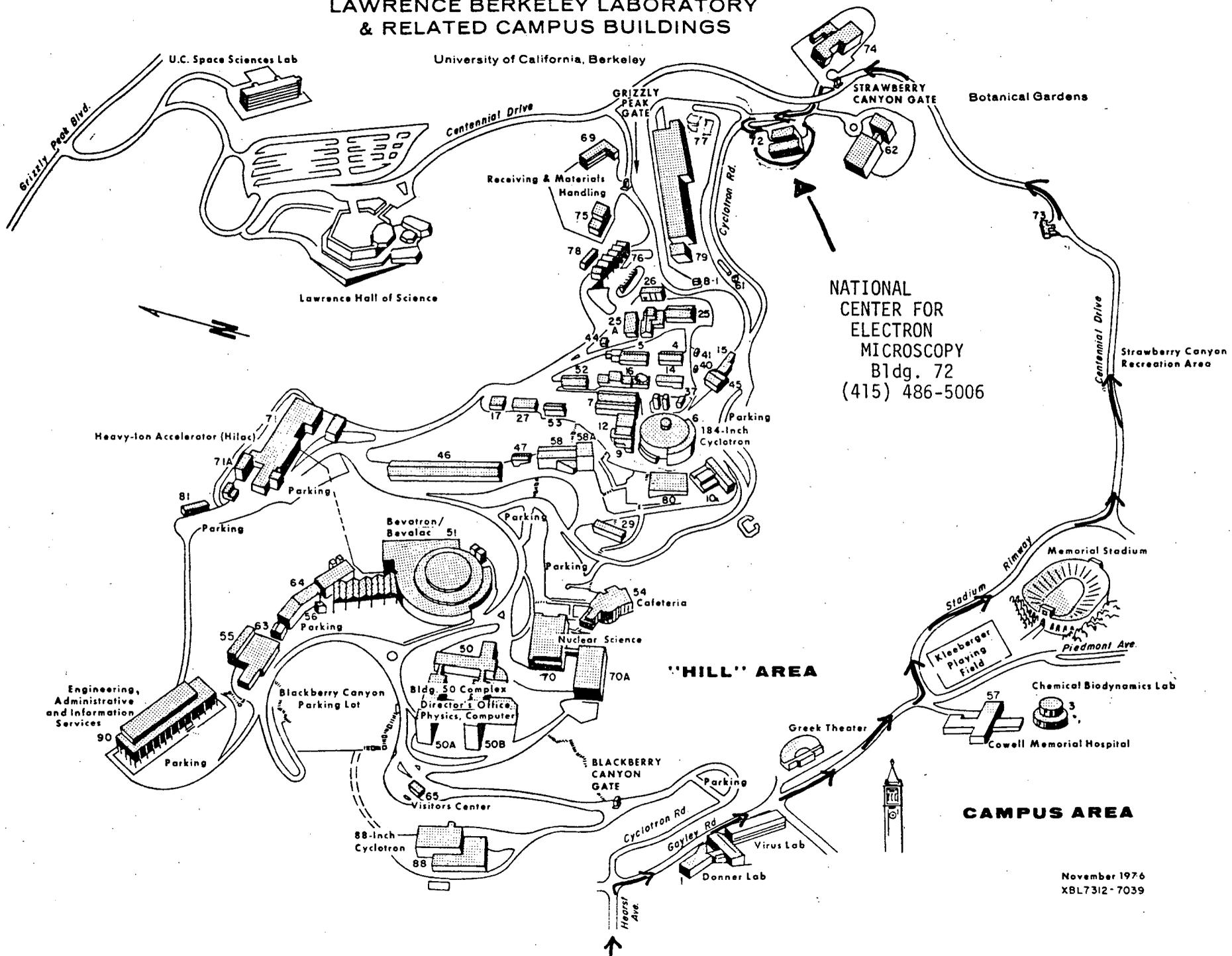
Take Hegenberger Road eastward from the airport for about 1.7 miles to Highway 17 (Nimitz Freeway). When approaching the Highway 17 intersection, bear right and watch for signs pointing to the turnoff to downtown Oakland.

Then go northwest about 8.5 miles on Highway 17, bearing right, to the intersection with Interstate 80 (Eastshore Freeway). Go north on it for about 2.4 miles, again bearing right, and take the University Avenue turnoff (right) into Berkeley. Stay on University Ave., about 2.6 miles, to its eastern end at Oxford St. (western side of U.C. campus), bearing left.

Turn left onto Oxford St. and, bearing right, take the first right turn onto Hearst Ave. and follow it up the hill to ~~LBL (Hearst Ave. becomes Cyclotron Road before the entrance to LBL).~~ *

From the east end of University Ave. to LBL is about 1 mile.

LAWRENCE BERKELEY LABORATORY & RELATED CAMPUS BUILDINGS



November 1976
XBL7312-7039

National Center for Electron Microscopy, Berkeley
 Research Summary Form

MMRD, Bldg. 72
 University of California
 Lawrence Berkeley Laboratory
 Berkeley, CA 94720
 (415) 486-5006

Title of Project: _____

P.I./Reseacher	Address	Phone Numbers		Please check appropriate box		
		Office	Home			
_____	_____	_____	_____			
_____	_____	_____	_____			
_____	_____	_____	_____			

Request to operate microscope _____

Request instruction to operate microscope _____

Will not operate microscope _____

Source and duration of project funding: _____

Estimate of Facility time required (no. of 8-hr. shifts) _____

Preferred HVEM*
 ARM 1000
 EM 1500
 HU 650

1st Preference _____
 2nd Preference _____

Scheduling Requirements:
 Earliest possible starting date: _____
 Latest possible starting date: _____
 Other time requirements: _____
 Anticipated completion date: _____

Special Equipment Requirements (e.g. stages, environmental cell, gases, etc.): _____

* Although the 1st preference will usually be obvious, please check 2nd preference and also indicate if preliminary work can be carried out on the HU 650.

NOTE: We, the investigators named above, have read, have understood and will comply with the operational requirements of the Facility as set forth in the Users' Guide.

Signature _____ Date _____

National Center for Electron Microscopy, Berkeley
 Schedule of Use

MMRD, Bldg. 72
 University of California
 Lawrence Berkeley Laboratory
 Berkeley, CA 94720
 (415) 486-5006

ARM 1000 _____
 EM 1500* _____
 HU 650* _____

Title: _____

Name: _____ Week of: _____

PLANNED			ACTUAL
Day/Date			
1st Preference	1st Alternative	2nd Alternative	Day

* Note Special Equipment Needs:

Environmental Cell _____

Gas _____

High Resolution Pole Piece _____

This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.