6.3 SCANNING ELECTRON MICROSCOPE FACILITY

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6.3.1 SCOPE

This section describes EML's scanning electron microscope (SEM) facility. The physical layout of this facility is shown in Figure 6.4. The SEM is used to: 1) analyze aerosols collected from the troposphere and the stratosphere (Kromidas et al., 1996); 2) characterize laboratory-generated aerosol; and 3) analyze biological samples.

6.3.2 SAMPLE PREPARATION ROOM

The sample preparation room is maintained under clean-room conditions using high efficiency particulate air (HEPA) ceiling filters. The air is recirculated and controlled with a system that is independent of the building air delivery system. There are two Class-100 benches to provide additional cleanliness for sample preparation and for the preparation of sampling equipment. The first, an exhausting laminar bench (Advanced Purification Inc., Hauppauge, NY 11788; Model G68-PD) allows the use of various solvents and provides a Class 100 environment for equipment setup and preparation of biological samples. The second bench (Laminar Flow Inc., Ivyland, PA 18974; special order) is a recirculating vertical laminar flow system with two cartridges that contain molecular sieve 5A and activated charcoal. These cartridges remove substances from the airstream that may interfere with aerosols. Such interfering substances include ammonia, water vapor, and organics.

6.3.3 MICROSCOPE ROOM

The microscope room is also maintained under clean-room conditions using HEPA ceiling filters.

6.3.3.1

SCANNING ELECTRON MICROSCOPE

The SEM is an AMRAY-1820 (AMRAY, Bedford, MA 01730) and has a 150-mm specimen chamber and a resolution of 5 nm. The SEM may be operated at low voltages (~1 kV), thus allowing imaging of samples without prior preparation. Microprocessor controlled operations permit automatic image focusing and stigmation and provides push button digital magnification from 25X- 400,000X at a 12 mm working distance. However, just how much of this magnification is usable depends heavily on the contrast characteristics of the sample. The SEM is supplied with a solid-state backscatter detector. This detector produces images which emphasize maximum atomic number differences. The resulting image shows contrast differences that can be related to differences in trace element composition. The SEM is also supplied with an X-ray/imaging system (described below). This system controls the sample stage automatically and thus allows for unattended X-ray analyses, imaging, and sample characterization of more than 100 different fields.

The whole microscope assembly is isolated on a Micro-g vibration isolation table (TMC, Peabody, MA 01960).

6.3.3.2

MICROANALYSIS SYSTEM

The microanalysis system used in conjunction with the AMRAY 1820 SEM is the PGT IMIX system with a two position light element Si(Li) energy dispersive spectrometer (Model 4200 EDS, Princeton Gamma-Tech, Princeton, NJ 08540). The PGT IMIX system is equipped with:

1. An ultrahigh resolution 19-in color display.
2. Dec 11/73 high performance processor with floating point.
3. SPARC station 1+ (SUN Microsystems) with a built-in, 3 1/2" floppy disk drive, external CD ROM drive, and a 1.27 gigabyte Fujitsu drive.

4. Two thousand channel data collection system.
5. Pulse processing electronics.
6. EDS X-ray analysis software.
7. Qualitative and quantitative analysis software.
8. Chemical classification software.
9. PGT beam control package.
10. Advanced feature analysis package.
11. Mega plane option (800 x 1040 pixel image).

The microanalysis system allows detailed chemical and size classifications of samples. In the windowless mode, the model 4200 EDS allows the analysis of X-rays with energies down to 700 eV. In this position, detection of B, C, N, and O is possible, as well as enhanced detection of F, Na, Mg, and Al. The PGT beam control package allows automated analysis of specific points on a collection surface by controlling the scan coils of the microscope column. Up to 300 points can be automatically stored for later use.

The PGT imaging system is connected to the Tissue Culture Facility via a video cable that is attached to a CCD video camera and can capture images from the optical microscopes.

6.3.3.3

SAMPLE CRITICAL POINT DRYING AND COATING EQUIPMENT

The sample coating equipment consists of a Denton high vacuum evaporator (Denton Vacuum, Inc., Cherry Hill, NJ 08003), an EFFA Carbon Coater (Ernest E. Fullam Co., Latham, NY 12110; Model No. 12560), and a sputter coater (Polaron, Warrington, PA; Model E5000). The sputter coater is supplied with platinum, gold, and silver targets. The carbon coater and sputter coater, plus an optical microscope are housed in a vertical flow class 100 laminar clean bench (Laminaire Corp., Rahway, NJ 07065; Model DWS-630).

Critical point drying of biological samples is accomplished via the Balzers CPD 030 using acetone as the transfer medium and liquid CO_2 as the transition liquid. The CPD 030 is located in the Sample Preparation Room with access to the exhaust laminar flow bench.

6.3.4 QUALITY CONTROL

When using an SEM, several things should be kept in mind. The sampling process must be conducted very carefully to provide the microscopist with a representative sample of the material of interest. Many methods of sampling introduce bias in that their collection efficiency varies with particle size. The internal environment of the microscope should also be taken into account. In order to function, the column is kept at a high vacuum (10^{-5} Torr) and there can be large amounts of heat generated in the sample by electron bombardment. These conditions can alter or destroy susceptible samples. Contamination is always a concern. Considering the size of the particles of interest, unless strict sample handling procedures are followed, contributions from contaminating sources may outweigh the sample material.

REFERENCE

Kromidas, L., and R. Leifer
"An Innovative Application of a Commercially Available Double-sided Adhesive for the Collection of Aerosols by Impaction"
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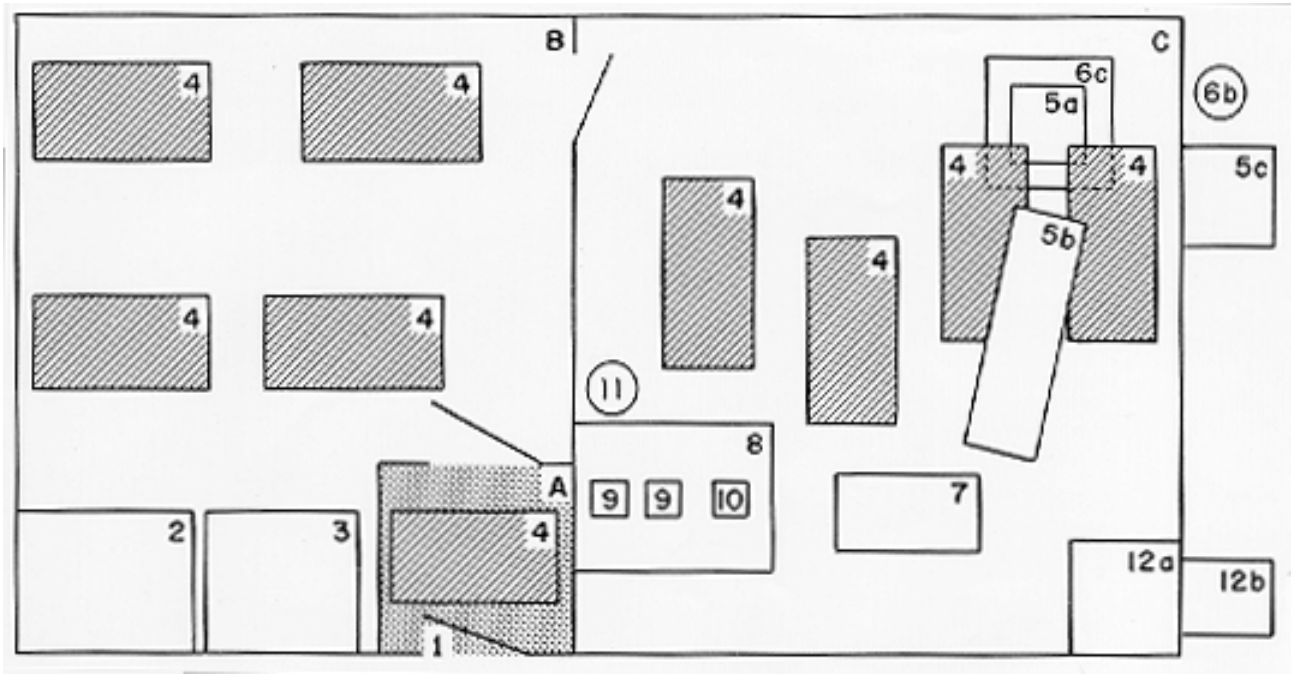

Figure 6.3 SEM facility: A) clothing change room; B) sample preparation room; C) SEM room. Areas in rooms: 1) sticky floor mat; 2) exhaust laminar flow bench; 3) recirculating laminar flow bench; 4) HEPA ceiling filters with blowers; 5a) column and stage; 5b) SEM electronic console; 5c) SEM vacuum, air and water support module (equipment placed outside microscope room); 6a) Micro-g vibration table; 6b) air supply for Micro-g table; 7) PGT microanalysis system; 8) vertical laminar flow bench; 9) particle coating devices; 10) optical microscope; 11) tank of Argon; 12a) Denton high vacuum evaporator; 12b) evaporator and coating device vacuum, air and water module.