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AEM of EXTRATERRESTRIAL MATERIALS

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NASA CONTRACT NAS 9-16491

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AEM of Extraterrestrial Materials
Quarterly Report
Final Quarter

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NASA Contract No. NAS9-16491
Introduction

The final quarter of the contract period has involved additional scientific presentations and publications, the development of a database for cosmic dust particles and the acquisition of additional data on extraterrestrial materials. A brief outline of the scientific and technical activities for this period is given below. This is followed by a summary of activities and major achievements during this contract period. Copies of publications for the 1981-1982 fiscal period are also appended.

Technical/Laboratory Status

The JEOL 100 CX electron microscope suffered a number of minor repairs during this period. These repairs included cleaning the upper condenser pole-piece and gun chamber, replacement of electronic relay switches and minor vacuum improvements. Cleaning of the microscope was necessary after changing out pole pieces, as the LaB₆ gun was incorrectly used. Tests of the analytical system were undertaken. These tests involved (1) a resolution test of the EDS detector, (2) monitoring beam stability with a recently installed faraday cup, (3) SEM magnification calibration (and resolution) after modifications and (3) trial calculations of elemental K factors for their film analysis. A PGT System IV analyser was installed with the JEOL 100 CX. The laboratory dark room has been rearranged to allow use of a Durst 138S enlarger. Minor errors in the packaging and configuration of the enlarger (as received) were rectified. In the area of specimen preparation, the thin section laboratory has successfully prepared a potted doubly polished section of a 20 um glass bead using Crystalbond 509 adhesive. This is an important step in the preparation of polished specimens of stratospheric dust.
Scientific Status

A considerable amount of effort has been spent on the cosmic dust project. With the help of a summer student, all relevant data on processed cosmic dust particles has been entered onto a retrievable data base system using the PDP 11/45. A total of 672 particles are currently listed in the general data base file which contains information on location, size, color, luster and qualitative chemistry. In addition, each particle in this file has been classified by a refined, logical classification scheme based primarily upon morphology and bulk chemistry. A second data base file containing quantitative bulk chemical analyses has also been constructed. This data base has already been used to investigate size distributions within certain classifications of stratospheric dust particles.

A chondritic aggregate specimen from Don Brownlee's collection has been studied using the AEM. This specimen is of interest because it seems to contain unusual minerals, perhaps even layer silicates. The nature of the minerals is still not completely determined, but layer silicates are not yet excluded from consideration.

The paper entitled "Analytical electron microscopy of extraterrestrial stratospheric particles" was presented at the 40th Electron Microscopy Society of America Meeting in Washington, D. C. Another paper titled "Al-Prime Particles in the Cosmic Dust Collection: Debris or not Debris?" was presented at the Meteoritical Society Meeting in St. Louis. A second paper on the Cosmic Dust Curatorial Facility was read on behalf of Uel Clanton and Jim Gooding at the St. Louis meeting. At least two other publications resulting from direct data collection or collaboration within the cosmic dust project have been submitted by NASA PI's. Unfortunately, it appears that neither authorship nor acknowledgement was extended on these publications. It is to be hoped that such unethical behavior will not occur again.
Support was given Dr. John Bradley to travel to JSC and present an excellent seminar on his recent observations on chondritic aggregates. Considerable discussion was generated during Dr. Bradley's visit and a collaborative abstract between the University of Washington, Washington University and the JSC groups has been accepted for the International Conference in Cometary Exploration in Budapest, Hungary.

The collection of ash from the El Chichon eruption in the stratosphere has caused a shift in the electron microscope laboratory activities. A number of samples have been examined in a preliminary fashion and this has resulted in the presentation of a paper at a workshop in San Francisco. An abstract has been submitted to the American Geophysical Union Fall meeting on El Chichon dust. Continuation of the work on shocked layer silicates has also resulted in the submission of an abstract for the Fall AGU meeting.

Finally, data collection for Volume 3 of the Cosmic Dust Catalog is complete, a patent application titled "Dedicated Specimen Holder for Spectral Contamination-Free Analytical Electron Microscopy" is submitted and tests on the degree of contamination in dealing with holey carbon films are in progress.

**Summary**

Repairs to the electron microscope dominated this past quarter's activities. However, two papers have been presented at meetings, three other abstracts have been submitted for presentation and a cosmic dust data base has been established. A summary of the year's activities follows in the next section.
Activities for 1981-1982 Contract Period

In the following section a brief outline of major achievements in the past year is given. This is followed by an addendum which contains copies of the first three quarterly reports, a list of publications for the contract period and sample copies of most of those publications.

A major portion of effort over the past year has been devoted to the cosmic dust collection. This has involved two separate activities: (a) cooperation with the Preliminary Examination Team (PET) for the Cosmic Dust Collection Program and (b) scientific studies on allocated cosmic dust particles. PET procedures are now well developed and part of laboratory routine. As a result of these PET procedures, and the subsequent data collection, three volumes of the Cosmic Dust Catalog have been produced and distributed to the scientific community.

Instrumentation

AEM: The modifications to the JEOL 100 CX outlined in the previous reports are now completed. These modifications have eliminated (from EDS analyses) any spectral contamination which may be attributed to the electron microscope column and environs. This was achieved by inserting special apertures within the upper column of the electron microscope and by modifying the specimen chamber components. New specimen holders for both SEM and TEM analysis were designed and built. Examples of the success of these modifications were informally presented at the recent Electron Microscope Society of America Meeting. The first published examples of the "spectral clean-up" will be evident in Volume 3 of the Cosmic Dust Catalog. There is currently only one other laboratory in the country which can demonstrate similar spectral cleanliness in a JEOL electron microscope.
The above modifications, in concert with improved detector collimation, newly developed software and a faraday cup, have provided us with a comprehensive quantitative analytical capability. Preliminary tests have been completed on thick flat specimens and single particles using both standard minerals and cosmic dust particles. The results to date are promising, and suggest that reliable quantitative analyses on cosmic dust particles are possible. Thin film standards (including samples used in thick flat analyses) have been prepared and are currently being used to calculate elemental k factors for the JEOL 100 CX. These k factors will then be used to analyze crushed chondritic aggregates as well as ion thinned meteorites and (perhaps) cosmic dust particles.

HRTEM: An ultra-high resolution pole-piece for use with a side-entry goniometer has been installed and tested on the JEOL 100 CX. Images obtained from this pole-piece were slightly better than advertised by the manufacturers (e.g., 3.4Å fringes in two directions with minimal beam tilt). Preliminary studies have been made on crushed chondritic aggregates as well as terrestrial samples.

Sample Preparation

Techniques for thin sectioning of fragile and difficult samples have been developed. These techniques include sections of carbonaceous chondrites as well as cosmic dust analogues. Ion-thinned samples of standard minerals, carbonaceous chondrites and terrestrial layer silicates have been prepared.

Cosmic Dust Particles

With the arrival of the first cosmic dust particles from the JSC collection, considerable effort was spent in devising satisfactory methods for examination and documentation. These methods soon formed the basis for the development of a data base on the Cosmic Dust Collection. A comprehensive data base containing information from a variety of sources is now an integral part of the cosmic dust project. This data base, containing optical, chemical and morphological descriptions of all proc-
essed JSC cosmic dust particles, is updated regularly. The data base also contains a revised classification scheme and permits easy retrieval of data for comprehensive studies of the collection.

Preliminary quantitative analyses have been made on 12 allocated spheres. These include a variety of sphere types, for example, aluminum spheres, aluminum prime spheres, iron-nickel spheres and calcium-aluminum-silicate spheres. These sphere analyses have shown that the particle analysis program is effective and that some sphere types may be considered single minerals.

Semi-quantitative analyses have also been made on single mineral grains from a disaggregated chondritic aggregate. The predominant types of minerals in this specimen are enstatite, olivine, pyrrhotite and possibly pentlandite. TEM and thin film analyses are also underway on an unusual chondritic aggregate provided by John Bradley and Don Brownlee.

**Meteorites**

Ion-thinned samples of Leoville and Murchison have been prepared and preliminary maps of the sections have been made. Other meteorite samples from the Alan Hills and the Yamato collections have been acquired and are currently being prepared for analysis. In addition to the above studies of carbonaceous chondrites, an investigation of the effects of shock on layer silicates has been initiated. This project is in conjunction with Philippe Lambert and Fred Hörz and is related to studies of the alteration processes of carbonaceous chondrite matrices. Using the AEM the shocked and unshocked mica phases in a meteorite analog sample have been analyzed. The shocked mica transforms to a glassy phase which retains the basic chemistry of the original crystalline mineral with the exception of water loss and minor amounts of volatile elements. Similar studies are planned for 1:1 layer silicates (e.g., antigorite).
Summary

A total of nine abstracted papers have been presented at scientific meetings and three others have been submitted for presentation. Three papers have been published or are accepted for publication, two others are pending acceptance. Three catalogs have been completed and a patent application has been made.
ADDENDUM

(Mackinnon Consulting Services; Contract No. NAS 9-16491)


Introduction

The first quarter of the current contract period was characterized by considerable technical advances, despite delays and confusion on the part of outside support. Many objectives for this period were attained. Some were prudently discarded and others newly formulated. A brief outline of the technical and scientific activities for this period is given below. This is followed by projections for the next quarter and a summary statement.

Technical/Laboratory Status

The primary activity within the laboratory has been the development of a complete analytical capability for the JEOL 100CX electron microscope. With the capable assistance of Drew Isaacs and GeorgAnn Nace, modifications to the electron microscope and to specimen handling and preparation techniques have been effectuated. The upper column has received a general "clean-up" in terms of "spurious" X-rays generated at various parts of the column. Extra apertures have been inserted within the condenser pole-pieces and at the condenser aperture in order to reduce the column X-ray flux. Test spectra before and after upper column modification indicate a significant decrease in the "spurious" X-ray count. The regions around the 100CX specimen chamber were then inspected for sources of "spurious" X-radiation. The guard tube below the objective lens was removed and sent to the machine shop for duplication in beryllium. Consideration was then given to the specimen holder and goniometer rod. Drew Isaacs initiated a replacement of the goniometer rod with an aluminum rod using a design from the microscopy group at Ann Arbor. Both the goniometer rod and the guard tube were left with NASA personnel for machining/duplication early in October. At the time of writing, neither product has been manufactured. A new specimen holder was designed in order to optimize ease of handling, the area of sample available for analysis and viewing,
and signal to noise ratio. A number of prototypes were then developed and shown to be effective. Rough plans were given to outside support for re-drawing in late October. The re-drawn plans were recently sent to the manufacturers for mass production.

A Gatan ion-mill arrived (after considerable delay) and was duly unpacked and operational within a half day of receipt. A number of small items were overlooked for the delivery. The ion beam coating devices were manufactured incorrectly, but were quickly replaced when Gatan were informed of the problems. A small 9x monocular optical microscope (for viewing into the milling chamber) was also obtained. Spare parts for the guns and specimen holders were ordered.

JEOL have delivered the backscattered electron detector, but without the necessary power supply. In addition to the power supply, a pole-piece, a LaB6 gun and special tilt and rotate holders are yet to be delivered by JEOL. A new PGT System III EDS analyser was demonstrated (on the 100CX) by the manufacturer.

With the development of the first "in-house" planchettes containing stratospheric dust particles, considerable effort has been directed towards PET responsibilities. In order to prevent further uninformed debate within LAPST meetings in matters of electron microscopy, a short report (sub-titled "Wheel No. 246783") was prepared for the Curator. This report outlined basic electron microscope phenomena (such as radiation damage, specimen charging, resolution, etc.) in a theoretical treatment and in an empirical manner using test specimens and techniques common to the laboratory. It appears that the PET recommendations regarding SEM techniques for stratospheric dust particles will be followed.

Methodology and bookkeeping techniques for the PET and curatorial facility were discussed and formulated. At the time of writing, 4 planchettes (representing a total of 90 particles) have been examined in a preliminary fashion.

A series of computer programs for the calculation of projected potentials and
high resolution images has been obtained from the Regional Facility for HREM at Arizona State University. These programs are currently being converted for local use in order to enable a comparison between high resolution experimental and calculated images. This is a necessary capability for the interpretation of images from "unknown" materials.

A large poster on "HRTEM of Beryl" for educational and demonstration purposes has been compiled and is on display in the electron microscope laboratory.

**Scientific Status**

The obvious need for technical developments over the past quarter have slowed scientific progress somewhat. However, the arrival of the Gatan ion-mill has led to a rapid improvement in specimen preparation procedures. In this respect a number of outstanding scientific problems now await experimental work on the JEOL 100CX. Since the ion-mill has been installed, the following specimens have been prepared: four mineral standards (for thin film analysis and/or high resolution imaging), two meteorites (one achondrite, one carbonaceous chondrite) and eight ortho-, chain- or layer-silicate minerals.

A paper titled "Ordered mixed layer structures in the Mighei carbonaceous chondrite" has been accepted for publication. The final version has been sent to the editor of Geochimica Cosmochimica Acta. A paper titled "Diffraction studies of a plagioclase megacryst" was presented at the Geological Society of America Annual meeting in Cincinnati. Prior to this meeting the Mineralogical Society of America short course on "Amphiboles" was attended.

The GSA meeting proved useful in other respects. Two scientists indicated a desire to work in the electron microscope laboratory at JSC. After some discussion and further correspondence, information and application forms for NRC Fellowships were posted to both scientists. A technician and graduate student with Roger Burns at MIT indicated an interest in possible collaborative work on carbonaceous
chondrites. A total of nine finely powdered samples of meteorites were sent to MIT for Mossbauer spectroscopy experiments.

A preliminary assessment of the stratospheric dust particles assigned to the current research project with David McKay indicates that at least 50% of the flags contain terrestrial contamination. The 55 particles available for study have been broadly categorized into terrestrial, lunar, meteoritic, cometary and unknown types on the basis of chemistry and morphology. A simplified non-genetic classification scheme has also been applied to these particles. Particles which potentially fall into the extraterrestrial categories are currently being investigated in detail. An unnecessary delay in the assignment of these particles to the current research project has left us with very little scientific data to date.

In the meantime, other projects which have received some attention over the past quarter include a SEM/TEM study of mesostasis in a lunar soil (with Uwe Reimold) and an AEM study of quenched basaltic glasses (with Gary Lofgren and Michael Baker). The apparent mesostasis grains show an unusual lamellar and/or angular morphology using SEM. The basaltic glasses reveal areas of crystalline material—primarily pyroxene and feldspar, with minor amounts of layer silicate. There appears to be a compositional zonation within glass adjacent to plagioclase grains. The zone is approximately 1000 Å wide and primarily shows a strong variation of Al with minor variations of Fe and Mg. This zonation can be interpreted in terms of equilibrium and metastability at the glass/plagioclase interface.

Projections

It is anticipated that the second quarter will include a continuing effort in the examination of stratospheric dust particles. This effort will be twofold—as part of the PET/curatorial workload, and as part of the current research project. Both of these activities will be directed towards publications within the next
quarter. Final modification will be made to the JEOL 100CX when appropriate machined parts are returned and when manufacturers supply the necessary items on order. This should occur during the second quarter. When the 100CX is finally modified a series of tests (using specimens already prepared) will be performed. Other science will be performed on a "when available" basis.

**Summary**

Many technical difficulties in the day-to-day operation of the electron microscope laboratory have been overcome. The laboratory can currently function in an "almost state-of-the-art" mode. Two publications have entered their final phase, ten specimens have been prepared for study, four stratospheric dust particle planchettes have received preliminary examination and two minor projects have received some attention. There is more to be done.
AEM of Extraterrestrial Materials

Quarterly Report

Second Quarter

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NASA Contract No. NAS9-16491
Introduction

The second quarter of the contract period has involved a number of scientific publications, a number of technical delays and a focussing of proposed activities for the final two quarters. A brief outline of the technical and scientific activities for this period is given below. This is followed by projections for the next quarter and a summary statement.

Technical/Laboratory Status

For a large part of this second quarter the JEOL 100 CX was inoperable due to major repair and maintenance procedures. These procedures coincided with the arrival of many additional parts which had been on order. Consequently, the backscattered electron detector and a new penning gauge were installed after a major clean-up of the column regions. Efforts to install and test the new ultra-high resolution polepiece have again been foiled by a stage part damaged during transit. A replacement part is being sent from Japan and will delay high resolution work until the next quarter. With regard to "X-ray clean-up" procedures, the manufacture of a beryllium guard tube has been unsuccessful. This is due in part to the brittle nature of the metal, and the ambitious nature of machineshop personnel. The aluminum goniometer rod has been in the care of Drew Isaacs and is near completion. Other specimen area modifications are complete. The new specimen holder design was submitted for patent application. A bulk supply of these holders still awaits action from the appropriate NASA scientist. In addition to the basic microscope down-time, there have been persistent and recurrent problems with the PGT energy dispersive analyzer.

As part of the continuing PET committment to the cosmic dust program, a further 13 planchettes have undergone preliminary examination. This represents a total of over 300 particles completed and ready for publication in the cosmic dust catalog. The second volume of the catalog is expected to be published this coming
quarter. A file of publication quality "mug shots" of selected cosmic dust particles has been established for access by Division scientists.

Scientific Status

The down-time in the laboratory has coincided with periods of activity in the publication of basic scientific results. A major commitment towards the publication of Volume 1 of the Cosmic Dust Catalog was completed on time. In addition, three papers were presented on the cosmic dust project. One paper, titled "Electron microscopy of fine-grained extraterrestrial materials," was presented at the Seventh Australian Electron Microscopy Conference in Australia. This was a very well-attended conference (it being the 25th anniversary of the first paper on the Cowley-Moodie multi-slice approximation) and many useful data were exchanged. A publication of this cosmic dust/meteorite paper will appear in the journal "Micron." Another paper was presented at the 13th Lunar and Planetary Science Conference in Houston. The title of the paper was "Electron microscopy of stratospheric particles." The third paper was also presented (by Uel Clanton) at the Lunar Science Conference. This paper (title: "Possible comet samples: The NASA cosmic dust program") described the curatorial activities (including PET) at JSC. The 13th Lunar Science Conference was notable for the considerable discussion generated on cosmic dust, and for a great variety of visitors to the electron microscope laboratory. These visitors included many of the PI's still actively involved in extraterrestrial research, in addition to two Russian scientists who attended the conference to report on their newly-acquired Venus data. Two other papers on cosmic dust have been submitted to the Spring AGU meeting (in Philadelphia) and the Proceedings of the 40th EMSA (in Washington, D. C.).

A manuscript titled "Ordering in Ni0-Mg0 solid solutions," coauthored with P. K. Davies and A. Navrotsky was submitted to the Journal of the American Ceramics
Society. A second manuscript titled "Subliquidus glass-glass immiscibility along the albite-diopside binary join," co-authored with D. J. Henry, I. Chan and A. Navrotsky was also submitted to Geochimica Cosmochimica Acta. A paper titled "A comparative study on the mineralogy, chemistry and chronology of the Apollo 16 crystalline impact breccias. Part II: Chronology," was presented by Uwe Reimold at the 13th Lunar and Planetary Science Conference in Houston.

The 13th Lunar Science Conference prompted much discussion on specimen preparation problems with cosmic dust particles. In response to this, a number of new techniques are under development. Initial success has been achieved with a mounting technique which may allow polished specimens of individual cosmic dust particles. While attending the Australian Electron Microscopy Conference contact was made with a technician willing to make ultra-thin sections of meteorites and layer silicates. As a preliminary step, two specimens (one meteorite, one layer silicate) have been sent to ANU for thin sectioning.

Projections

It is anticipated that in the third quarter the PET commitment to cosmic dust particles will continue. This effort should also include the installation of a new JSEM 35 electron microscope with attendant procedural changes. The analytical capability of the 100 CX should be fully operational and results on standard analyses and extraterrestrial materials will be available. These activities, and those of the past quarter, should result in additional publications in the third quarter.

Summary

Laboratory down-time has been significantly high this past quarter, although with beneficial results. Two manuscripts have been submitted for publication, four papers have been presented at conferences, Volume 1 of the cosmic dust catalog has been published, thirteen stratospheric dust particle planchettes have received
preliminary examination and specimen preparation procedures have received some attention.
AEM of Extraterrestrial Materials

Quarterly Report

Third Quarter

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NASA Contract No. NAS9-16491
Introduction

The third quarter of the contract period has involved a number of scientific publications and presentations, improvements in the instrumental capabilities of the microscope, and the acquisition of useful data for various extraterrestrial materials. A brief outline of the technical and scientific activities for this period is given below. This is followed by projections for the next quarter and a summary statement.

Technical/Laboratory Status

Modifications to the JEOL 100 CX during this period included a conical shaped collimator for the EDS detector, and a new bore for the specimen chamber. Tests for the optimum detector-specimen distance were also performed. After an initial false start and further delay, the ultra-high resolution pole-piece was installed. Other additions at this time included a new stage flange, an outer ACS and a LaB$_6$ gun. Resolution tests at the time of installation were almost satisfactory (3.4Å with tilted beam, one dimensional). After a few days, the machine performance improved (3.4Å, two dimensional). Tests on the new specimen holders for the UH pole-piece showed that they were unsuitable for analytical work. New holders will be designed to exploit the full potential of this latest modification.

Other laboratory-related activities included the extension of the "publication files"—an additional file containing slides for presentations and a display on cosmic dust for the laboratory foyer.

Scientific Status

Publications have again figured in the past quarter's activities. A paper entitled "Classification of the JSC Stratospheric Dust Collection" has been accepted for publication in the Journal of Geophysical Research. This paper presents an overview and preliminary assessment of the cosmic dust collection at
JSC to date. The paper entitled "Mineralogy of Extraterrestrial Particles from the Stratosphere" was presented at the American Geophysical Union conference in Philadelphia in May. Another paper entitled "Mineralogie und Chemie der Kristallinen Apollo 16 Impakttschmelz-gesteine" was presented at the Paneth Kollozium in Zurich in June. A paper on Al-prime particles has been submitted to the Meteoritical Society in St. Louis.

Two seminars on cosmic dust were presented during this quarter. The first was a current report on the cosmic dust program to local scientists in the Division of Earth and Planetary Sciences. The second presentation, of a more general nature, was to the Houston Area Microbeam Analysis Society at the University of Houston. In addition, manuscripts from the editors of the American Mineralogist and the Journal of Geophysical Research were critically reviewed.

Requests for meteorite samples were submitted to the Antarctic Meteorite Working Group in Houston, and to the National Institute for Polar Research in Japan. In addition, two meteorite samples, Cold Bokkeveld and Allende, were received from Carleton Moore in Arizona. Fragments of both these meteorites were sent to ANU in Canberra for thin sectioning. In this regard, a local company has been contacted in the hope that fast, reliable, and satisfactory production of thin sections can be implemented.

Research projects related to the cosmic dust/extraterrestrial project have also been pursued. These projects include an investigation of the El Chichon ash-fall in association with Chuck Wood and others, an AEM study of shocked micas with Phillipe Lambert, and a continuing study of terrestrial mixed-layer clays. While in the United Kingdom for a visa renewal, the HREM facility at Cambridge University was visited. A revised, updated version of computed image programs was obtained from the author, Michael O'Keefe.
Visitors to the laboratory included Jim Boland, an electron microscopist from Utrecht. Correspondence with Franz Reitmeyer in Utrecht resulted in his application for an NRC postdoctoral fellowship at JSC. A summer intern has been given assistance with a small-scale research project.

**Projections**

It is anticipated that in the fourth quarter the PET commitment will continue, the UH pole-piece will be replaced with a conventional pole-piece, and data acquisition and reduction will be performed on allocated cosmic dust particles. A bid will also be made to renew the current contract for a further financial year period.

**Summary**

Substantial modifications to the JEOL 100 CX electron microscope have been successful, although with some loss of user time. One paper has been accepted for publication, one abstract submitted for presentation, one paper has been presented at the AGU Conference, and Volume 2 of the cosmic dust catalog is published. Other peripheral projects have received considerable attention, while sample preparation continues.
Introduction

The final quarter of the contract period has involved additional scientific presentations and publications, the development of a data base for cosmic dust particles and the acquisition of additional data on extraterrestrial materials. A brief outline of the scientific and technical activities for this period is given below. This is followed by a summary of activities and major achievements during this contract period. Copies of publications for the 1981-1982 fiscal period are also appended.

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The laboratory dark room has been rearranged to allow use of a Durst 138S enlarger. Minor errors in the packaging and configuration of the enlarger (as received) were rectified. In the area of specimen preparation, the thin section laboratory has successfully prepared a potted doubly polished section of a 20 um glass bead using Crystalbond 509 adhesive. This is an important step in the preparation of polished specimens of stratospheric dust.