

Proposal

*Thermochronological Evolution of Calcite Formation at the
Proposed Yucca Mountain Repository Site, Nevada*

Submitted to the
Department of Energy

by

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ABSTRACT

This proposal addresses the question of whether or not fluids with temperatures in excess of normal ambient temperatures have infiltrated the Yucca Mountain repository site in the recent geologic past. Previous studies of fluid inclusions in open space calcite mineralization have produced conflicting observations and data that have been interpreted to indicate that either 1) rocks at the level of the repository site have been in a vadose zone environment since shortly after their deposition, or 2) thermal fluids have risen from depth, at some time in the recent past, and flooded the repository site. Studies conducted during this project will be directed towards answering the following questions.

1. Do populations of fluid inclusions, that indicate the recent influx of thermal waters into the repository site, exist?
2. If these inclusions are present, what minimum fluid temperatures do they indicate?
3. If present, when were these inclusions trapped, i.e., when did this influx(es) occur?
4. If an influx occurred, how widespread, within the repository site, was this influx?

To answer these questions, the following studies will be conducted.

Phase I: Collect approximately 200 samples throughout the Exploratory Study Facility and cross drift. Doubly polished sections will be prepared from each sample and examined for the presence of two-phase fluid inclusions with consistent liquid-vapor ratios.

Phase II: Each section will be characterized using petrography, trace element geochemistry, cathodoluminescence, surface imaging, and oxygen and carbon isotopes, so as to produce a "time map" for each section, which documents the progressive growth of the calcite. Two-phase fluid inclusion assemblages will be identified and located to determine inclusion origins, and determine relative ages of the assemblages based their origins and locations within section time maps.

Phase III: Conduct microthermometric studies to determine minimum homogenization temperatures and salinities of two-phase fluid inclusion assemblages, and salinities of liquid-only inclusions. Conduct crushing studies to determine internal pressures of vapor inclusions. Conduct other analytical studies identified during the project as being capable of identifying inclusion fluid compositions.

Phase IV: Select samples of calcite and opal for geochronological studies which will provide maximum and minimum ages for primary, two-phase fluid inclusion assemblages, and maximum ages for secondary fluid inclusion assemblages, with consistent, elevated homogenization temperatures. Prioritize samples based on inclusion origin, and inclusion location in the "younger" portion of the samples. Integrate U-Pb and uranium series dates with stable isotope results to further constrain inclusion ages.

Phase V: Publish.

Scientists representing both DOE and the State of Nevada, and an independent expert advisor will be invited to attend quarterly meetings, during which UNLV scientists will present all data. Goals for each meeting will include: 1) examining, evaluating, and reaching a consensus on work completed to date, 2) discussing, evaluating, and eliminating or adding viable hypotheses, 3) defining methods to test hypotheses, and 4) defining methodology for the next phase of data collection.

A major goal of this project is for UNLV, Nevada, and DOE scientists to reach and maintain a consensus at each step during the study. If this can be accomplished, we should be in agreement, and all be aware of the strengths and weaknesses of the data and conclusions, when the project comes to a close. With this in mind, a written document identifying those aspects of the research where there is unanimous agreement, as well as

those aspects of the study where consensus is yet to be reached, will be prepared at the conclusion of each quarterly meeting.

PROJECT SUMMARY

Description of the Problem

The proposed Yucca Mountain nuclear waste repository is unique among repository sites owing to its location, underground, above a deep water table. Because a future rise in the water table would adversely affect the integrity of the site, it is relevant to examine the geologic history of the region to ascertain whether the deep water table is a stable feature with a lengthy geologic history, or whether fluctuations in water table elevation occurred in the recent geologic past. Such fluctuations would indicate an increased potential for fluctuations during the regulatory lifetime of the repository.

Previous studies addressing this question have focused on open space calcite mineralization to determine whether it formed in an unsaturated, or saturated environment. Some researchers concur that calcite precipitated from downward percolating cold meteoric fluids in a vadose environment (Paces et al., 1996; 1997). Dublyansky (1998a; 1998b), however, reports the presence of two-phase, aqueous fluid inclusions in calcite, and interprets these inclusions to have precipitated from, and to have trapped upwelling hydrothermal fluids.

Review of Dublyansky's and other studies by four technical experts led to the recommendation that the timing of formation of the two-phase fluid inclusions be established. This proposal describes a study designed to examine calcite mineralization, constrain the timing of identified two-phase fluid inclusions with consistent elevated temperatures, and to determine the areal extent of such inclusions.

Specific questions that this proposal addresses are:

1. Do populations of fluid inclusions, that indicate the recent influx of thermal waters into the repository site, exist?
2. If these inclusions are present, what minimum fluid temperatures do they indicate?
3. If present, when were these inclusions trapped, i.e., when did this influx(es) occur?
4. If an influx occurred, how widespread, within the repository site, was this influx?

Project Summary

This project has been divided into five phases, during which the various component studies will be conducted. The tasks and activities in each of the phases are described and discussed in detail in the body of the proposal. A brief description of the activities in each phase is given below. Progress in each of the phases of the project will be summarized in quarterly reports, following meetings with State and DOE representatives, and a technical expert.

Phase I includes sample collection, doubly polished section preparation, and petrographic study of the sections to locate populations of two-phase fluid inclusions. An important part of this phase of the study will be to handle all samples so as to protect their integrity. Extreme care will be taken so that the samples are neither heated nor frozen during collection, transportation, section preparation, and storage.

Phase II is the sample characterization study, which involves identifying the growth history of sections prepared from each sample. The growth history will be determined by identifying growth zoning through the use of integrated petrographic and electron microprobe studies, supplemented by stable isotope studies. This portion of the project is critically important as it will provide "time maps" for each section, which will

govern the selection of sample sites for all micro analytical work, including fluid inclusion, stable isotope, and geochronological studies. Phase II also includes identifying and characterizing all fluid inclusion populations in each section, and determining the relative timing of inclusions, based on inclusion origin and location in the section.

Phase III is the fluid inclusion component of the study. During this part of the project fluid inclusion assemblages will be examined using microthermometry, crushing, and, possibly, other analytical methods. Studies will involve determining homogenization temperatures and salinities of two-phase inclusion assemblages, determining salinities of liquid-only inclusion assemblages, and examining vapor-only inclusions during crushing to determine internal pressures of inclusions. The locations, within the sections, of the inclusions will be noted. Additional qualitative or quantitative analyses may be identified and conducted during the study, to identify inclusion compositions.

Phase IV involves geochronologic studies to constrain ages of two-phase fluid inclusion assemblages. Opal and/or calcite collected from growth bands that bracket two-phase inclusion assemblages with consistent, elevated temperatures will be submitted for uranium series or U-Pb analyses. Sample material will be prioritized to evaluate primary and "young" inclusion populations. Absolute age determinations will be supplemented by traditional, and ion probe, in situ, oxygen and carbon isotope analyses, which correlate with calcite age. Micro samples for uranium series and U-Pb dating and traditional isotope analyses will be collected using a computer controlled micro sampling device, which has a sample resolution of 10 micrometers. Geochronologic data and oxygen and carbon isotopic analyses will be evaluated in concert with results from the fluid inclusion studies to constrain ages of two-phase fluid inclusions with consistent, elevated homogenization temperatures.

Phase V involves submitting, for publication, one or more manuscripts describing the studies, results, and final conclusions of this project.

Equipment Summary

Two pieces of equipment are required to conduct the described research. Instruments are briefly described below; detailed descriptions, justifications, and costs are provided in the body of the proposal.

The purchase of an electron microprobe is requested, so that the integrated petrographic and geochemical, cathodoluminescence, and surface imaging studies, required to characterize the temporal growth of the calcite samples, can be conducted. The in-house availability of this equipment is essential for the sample characterization study to be conducted in a rigorous and timely fashion, and to produce the quality results required for successful completion of the project. A detailed description of, and justification for, this equipment is provided beginning on page 21.

Purchase of Micromill, a micro sampling device designed for high resolution milling to recover sample powders for chemical and isotopic analysis, is requested. This instrument will permit precise sampling of individual, or a small number of growth zones, for geochronological and isotopic analysis. A more detailed description of this instrument is provided on page 23.

Personnel Summary

This study will be conducted by the principal investigator (PI) and a post doctoral fellow (PD). The PI is responsible for overseeing the successful and timely completion of the project, as outlined in this proposal. The PD will be responsible for conducting the scientific studies, as outlined, preparing quarterly reports, and for preparing manuscripts for publication on completion of the project. Responsibilities for both of these positions are described in detail beginning on page 14.

An electron microprobe technician will be employed to aid in the installation of the microprobe, to calibrate the instrument so that it is fully functional and meets QA

standards, and to aid in the collection of geochemical and cathodoluminescence data, and image analyses.

Two students will be hired to assist with the project. The first student will be employed full-time for two months, to assist the PD with sample collection and sample preparation for doubly polished sections. The second student will be employed half-time for the duration of the project to perform secretarial and other non-technical tasks associated with the project. Details for these positions are provided beginning on page 15.

An independent technical advisor will be employed to attend quarterly meetings to discuss and evaluate the project. This position is described more fully in the following paragraphs and beginning on page 17.

Nevada, DOE, and Independent Technical Expert Participation

This proposal addresses an issue which has been shown to be extremely contentious, but which is of significant relevance to the suitability of the proposed repository site. Previous studies have produced conflicting observations and interpretations, which have been questioned or rejected by those on "the other side" of the argument. There is little to be gained by conducting another independent study, which is likely to be rejected by a segment of the interested parties upon completion. It is, therefore, my goal to conduct this study with the participation of both the State and DOE, such that, at the conclusion of the study, the issue is resolved to the satisfaction of both the State of Nevada and DOE. To this end, I invite the State and DOE to designate qualified representative scientists to attend week-long, quarterly meetings, during which they will contribute to defining data collection methodology, view and evaluate data, and contribute to data interpretation and construction of hypotheses consistent with these data. An independent technical expert, whose role will be that of facilitator (and referee), will participate in these meetings. Meetings will begin with the presentation, by UNLV scientists, of all data collected since the previous meeting. Participants will then have the opportunity to question the researchers and examine all data, samples, doubly polished sections, and all other project-related materials. Group goals for each meeting will include: 1) examining, evaluating, and reaching a consensus on work completed to date, 2) discussing, evaluating, and eliminating or adding viable hypotheses, 3) defining methods to test hypotheses, and 4) defining methodology for the next phase of data collection. The technical expert is an authority on fluid inclusions and is familiar with the fluid inclusion database as it relates to the current controversy concerning the origin of calcite at Yucca Mountain, but has no connections to either the State of Nevada or to DOE. Thus, this person will have a unique perspective, and "some distance" from the project, that will allow him to facilitate group discussions, evaluate observations and interpretations, and referee disagreements among group members. Most importantly, his role will be to promote compromise on issues such as methodology, data interpretation, and development of hypotheses.

PROJECT DESCRIPTION

PHASE I: Sample Collection and Section Preparation

- Tasks:
1. Collect samples of all identified varieties of calcite encountered in ESF and cross drift outcrops.
 2. Collect samples from throughout the repository site so as to determine the spatial distribution of two-phase fluid inclusions.
 3. Prepare doubly polished sections using techniques which will not heat or cleave or otherwise damage samples.

4. Conduct a preliminary survey of the sections, searching for fluid inclusion assemblages (FIAs) of two-phase inclusions.

Procedures:

Approximately four weeks will be spent collecting samples at the Yucca Mountain repository site. To test for the presence of two-phase fluid inclusions from throughout the site, approximately 200 samples will be collected from the 7900 meter long Exploratory Studies Facility (ESF) and the recently constructed 2800 meter cross drift. This will provide for a sampling density of roughly one sample every 50 meters. Samples will be collected and labeled using established DOE procedures. All varieties of calcite, including calcite from lithophysal cavities, high angle fractures, flatter fractures, and breccias, will be collected in order to conduct a thorough search for fluid inclusion populations. Samples will be collected near fault zones and our sample sites will duplicate many of those previously selected by the USGS, DOE and the State of Nevada. In particular, we will duplicate sampling by Dublyansky, that yielded two-phase inclusions with consistent, elevated homogenization temperatures (Dublyansky, 1998b). Material selected from each sample site will include that which is most representative of the calcite present. We will also include material which contains uranium-bearing opal, identified with a black-light. Small samples of this opal have been previously dated using U-Pb and collection of this material will aid in constraining ages of opal-bearing zones in the samples.

It is essential to collect, transport, and store these samples such that they are not subjected to temperatures above 35°C or below freezing. Samples will be transported personally by the PI or PD and will be stored in the PI's laboratory in the geoscience building.

Doubly polished sections will be prepared by Petrographic Services in Montrose, Colorado. Mark Mercer, the owner of Petrographic Services, has nearly twenty years full-time experience making sections, and has owned this business since 1986. Mercer was highly recommended by the Kansas Geological Survey for his experience in making sections from limestone, dolomite and calcite for petroleum-related and diagenetic studies. Sections prepared for Kansas Survey personnel preserved delicate textural features and contained low temperature fluid inclusion assemblages, indicating that the sections had been carefully prepared and had not been heated.

Mercer has previously prepared polished thin and thick sections of Yucca Mountain calcite for U.S.G.S. personnel. Thus he is familiar with the material and has developed procedures for section preparation. Samples will be sawed using a water cooled diamond saw, and will be ground on a diamond wheel. Delicate samples will be impregnated using an epoxy that cures at room temperature. Polishing will be done by hand using an aluminum oxide grit, followed by polishing with a diamond compound on a nylon cloth. Sawing, grinding, and polishing will be done at slow speeds so as to minimize frictional heating.

Prepared sections will be examined under a petrographic microscope to identify those samples that contain two-phase inclusions with consistent liquid-vapor ratios. Cleavage chips from representative samples that contain two-phase inclusions will be examined to confirm that the samples have not been damaged during sample preparation. The presence of two-phase inclusions in cleavage chips (that have undergone no sample prep) will provide convincing evidence that the inclusion phase ratios represent natural conditions are not a result of damage during sample preparation. Two research quality petrographic microscopes with transmitted and reflected light capabilities are available in the PI's laboratory at UNLV. A second section will be prepared for each sample that contains two-phase inclusions, and will be used for analytical work which is either destructive, or which heats the section.

Phase I will require approximately four months to complete.

PHASE II: Sample Characterization

- Tasks:**
1. Identify visual and chemical zoning in prepared sections that reflects crystal growth. Methods will include: 1. petrographic studies to identify visually distinct regions or zones, and the presence of solid or fluid inclusions, 2. cathodoluminescence studies to locate luminescing and non-luminescing growth zones, 3. electron microprobe analyses to identify micro trace element-zoning, 4. imaging of three-dimensional sample surfaces to identify the nature of mineral contacts and zoning, and 5. laser ablation, induced coupled plasma mass spectrometer (LA-ICPMS) micro chemical analyses to further constrain chemical zoning.
 2. Identify all populations of fluid inclusions present in each section.
 3. Place initial relative temporal constraints on the trapping of identified fluid inclusion populations by 1) noting inclusion locations, with respect to zoning in calcite, and 2) identifying inclusion origins: primary, secondary, pseudosecondary, or unknown.
 4. Further constrain relative ages of inclusions using secondary ion mass spectrometer (SIMS) analyses to determine carbon and oxygen isotope ratios in calcite growth zones that bracket two-phase inclusions with consistent phase ratios.

Procedures:

Previous petrographic studies have documented the presence of a variety of calcite morphologies (Whelan et al., 1994; Whelan and Moscati, 1998; Dublyansky, 1998b), the presence, in calcite, of growth zones marked by semi-opaque inclusions including organic matter (Whelan et al., 1994; Whelan and Moscati, 1998) and fluid inclusions (Dublyansky, 1998b), and laminated opal (Paces et al., 1996). A stratigraphy of calcite and minor opal, identified in lithophysal cavities, exhibits old, blocky to thick-bladed, inclusion-rich calcite and thicker opal, which is overgrown by euhedral sparry, water-clear calcite, which locally forms thin-bladed to fan-shaped crystals, and late, clear to translucent, bubbly to finely laminated opal (Paces et al., 1996; Paces et al., 1997). In fractures, calcite forms sheet-like accumulations of sparry, equant crystals (Paces et al., 1997).

Petrographic studies will be conducted on sections containing two-phase fluid inclusions, to identify zoning indicated by these and other textural features. Textural characteristics which are common to older, intermediate, and younger calcite, as indicated by their relative location in the section will be noted. Samples will be examined to determine whether or not a correlation between inclusion populations and types of calcite exists.

Cathodoluminescence studies have shown that calcites exhibit alternating luminescing and nonluminescing zone pairs, 1 to 20 micrometers in diameter (Denniston et al., 1997; see their figure 4A). These paired bands, which contain trace variations in iron, manganese, and other elements, are important because they reflect growth zoning within the calcite crystals and will allow the relative ages of different sections of the calcite to be identified. Cathodoluminescence studies have revealed at least four major stages of calcite deposition, in which different styles of growth banding are revealed by luminescence (Whelan et al., 1994). Zoning varies from early, usually non-luminescing or faintly luminescing bands, to intermediate stage non-luminescing with bright yellow bands, to young, thin and weakly luminescing bands (Whelan et al., 1994). Local discontinuities in luminescent bands mark hiatuses in calcite precipitation, and regions where competing crystals have grown together.

Cathodoluminescence studies will be conducted on all samples containing two-phase inclusions, and will be integrated with petrographic studies to provide the primary control for identifying the growth sequence of the calcite crystals. As

cathodoluminescence analyses heat thin sections, these analyses will be conducted on a second section prepared from samples containing two-phase fluid inclusions. Results from these studies will contribute to identifying regions in the sections which will be analyzed using micro chemical techniques.

Previous studies have shown that variations in minor and trace elements exist in shallow calcites (Whelan et al., 1994; Denniston et al., 1997). Manganese and REEs exhibit small but distinct variations; iron and strontium exhibit greater ranges of concentrations, 15.2 to 200 ppm and 5.6 to 167 ppm, respectively (Denniston et al., 1997). Electron microprobe analyses and elemental maps will be used to identify iron and strontium zoning. As zoning of primarily iron and manganese is responsible for luminescence in these calcites, chemical zoning should correlate with, and confirm zoning indicated by cathodoluminescence. Chemical analyses will provide zoning information for samples or regions which do not, or poorly luminescence.

Important textural relationships distinguishing various generations of calcite and opal were documented with high resolution, three-dimensional images of sample surfaces (Paces et al., 1996). These images illustrated, for example, sharp crystal faces and edges at calcite-opal contacts, indicating that surfaces represented terminations of crystal growth and not dissolution. We will conduct similar imaging studies which will supplement petrographic and geochemical information on crystal growth and zoning.

Laser ablation-induced coupled plasma mass spectrometer (LA-ICPMS) analyses provide a method for precise, in situ, micro geochemical analyses (Sylvester, 1997). This technique, which has an analytical spot size of 20 micrometers, will be used to determine the chemistry of individual, or a small number of, calcite growth zones. As LA-ICPMS has lower detection levels than the electron microprobe analyses, This technique will be used to supplement microprobe results in trace element poor regions of the calcite samples.

After the growth history of each section has been determined using petrographic and geochemical methods, all sections will be inspected to identify fluid inclusion populations that are present, to determine the relative locations of each inclusion population with respect to the growth history of the section, and to determine the origin of these inclusions. Particular attention will be paid to populations of two-phase fluid inclusions that exhibit consistent liquid-vapor ratios. These observations will be critical in identifying inclusions for the microthermometric study.

Carbon isotope ratios (Whelan and Moscati, 1998) show a clear trend from higher values for older calcites to lower values for younger calcite. The range of values obtained for old, intermediate, and young calcites is great enough (Whelan and Moscati, 1998; see their figure 2), however, that an isotopic ratio cannot be used to unequivocally identify the relative age of the calcite sample. Nevertheless, their data show that calcites older than 4 Ma have $\delta^{13}\text{C}$ values larger than -4 per mil; calcites younger than 1 Ma have $\delta^{13}\text{C}$ values less than -4.5 per mil (Paces et al., 1997). These data are from calcite micro samples of less than 0.1 mg collected from polished surfaces of petrographic sections. Although these samples are very small, it is likely that the spread in the data results from samples containing multiple growth zones. We will use secondary ion mass spectrometer (SIMS) analyses to determine in situ, carbon and oxygen isotope ratios in calcite. This method allows analysis of a spot, 15 to 20 microns in diameter, and will minimize the number of growth zones evaluated in a single analysis. Such analyses should restrict the range of isotopic ratios for old, intermediate, and young calcites. If it can be clearly established that calcites of different ages exhibit discrete ranges of isotopic signatures, the correlation between isotopic signatures and radiometric ages (Paces et al., 1997; see their figure A4) can be refined, and isotopic signatures can be used to place important temporal constraints on calcite growth zones that bracket two-phase fluid inclusions.

SIMS analytical work will be conducted in two stages. Initially, data will be collected on traverses along the length of the calcite section, or parallel to the growth of

the crystals, and along growth zones in calcite. These data will be used to identify ranges of isotopic ratios that represent relative ages during crystal growth, and to establish isotopic consistency for individual growth zones. Data will be correlated with visible zoning, calcite crystal morphology, and other textural features identified during the petrographic study. Secondly, if it can be established that restricted isotopic ratios correlate with relative calcite age, isotope ratios will be determined for calcite growth zones that border two-phase fluid inclusions exhibiting consistent and elevated homogenization temperatures, providing important relative temporal constraints for these inclusions. Upon completion of geochronologic dating (described below) isotopic ratios will be evaluated in light of absolute ages determined for calcite and opal zones in the sections and absolute age constraints may be placed on ranges of isotopic ratios.

Phase II will require approximately six to twelve months to complete. The length of time will be principally determined by the number of two-phase fluid inclusions assemblages with consistent liquid-vapor ratios found during phase I of the study.

PHASE III: Fluid Inclusion Studies

- Tasks:**
1. Select fluid inclusion assemblages (FIAs) of two-phase liquid-rich inclusions for microthermometric studies to determine inclusion homogenization temperatures and fluid salinities.
 2. Select FIAs of liquid-only inclusions for freezing studies to determine fluid salinities.
 3. Select vapor-only inclusions which are distributed in old, intermediate, and young calcite, for crushing studies to determine approximate internal pressures of inclusions.
 4. Conduct other qualitative or quantitative analyses identified during the course of the study as suitable for identifying the compositions of contained fluids.

Procedures:

Previous studies have shown that the most abundant fluid inclusions in calcites from the ESF are liquid-only inclusions. Vapor-only inclusions are less abundant, but are commonly present. Two-phase fluid inclusions are present, but their occurrence is rare (Roedder et al., 1994; Dublyansky, 1998b). Roedder et al. (1994) concluded that the abundance of liquid-only and vapor-only inclusions provides evidence for trapping of either liquid water (liquid-only inclusions) or air (vapor-only inclusions) in a vadose zone environment. They further believe that the two-phase inclusions they observed formed under a higher geothermal gradient in oldest calcite, trapped a mixture of liquid and vapor, were stretched during a transient, elevated geothermal gradient, or were stretched while drill core containing the inclusions was exposed to elevated temperatures (Roedder and Whelan, unpublished manuscript). Dublyansky (1998b) presented homogenization data for seven samples in which two-phase fluid inclusions show consistent homogenization temperatures. Most inclusion homogenization temperatures are less than $\sim 50^{\circ}\text{C}$, and modes are commonly at 30° to 35°C ; however, two samples contained inclusions with consistent homogenization temperatures that reached 61° and 75°C . The inclusion assemblage with a maximum temperature of 75°C is described as being located in the paragenetically older portion of the calcite; the location of the other inclusion assemblage is not given. Dublyansky (1998b) concludes that these seven samples provide evidence for trapping of a single-phase fluid with an elevated temperature. The proposed study will test these hypotheses, and will examine other hypotheses which arise during the course of the investigation.

A fluid inclusion assemblage, as defined by Goldstein and Reynolds (1994), is a group of fluid inclusions that formed at the same time or at the same conditions, and

along a growth zone in a single crystal or along a single healed fracture. In order to correctly interpret fluid inclusion data, it is essential to measure many fluid inclusions within an FIA (Goldstein and Reynolds, 1994), as only through FIA evaluation is it possible to discern variability of the data which will indicate the possibility of reequilibration, necking, or immiscibility. Consistency of data among inclusions of different sizes and shapes within an FIA indicates trapping of homogeneous fluids and that assumptions of constant mass and volume are accurate. The proposed study will be conducted using the FIA philosophy (Goldstein and Reynolds, 1994) and microthermometric and other analyses will be conducted only on inclusions fulfilling FIA criteria.

Sections identified during sample characterization as containing two-phase inclusions will be thoroughly reexamined and all two-phase FIAs will be noted for evaluation. As previous studies have indicated that two-phase inclusions are rare, we anticipate conducting microthermometric analyses on all such FIAs identified. Vapor bubbles in these inclusions have been previously shown to be small; thus heating studies will be conducted prior to freezing studies, to eliminate the possibility of stretching during freezing. Inclusions will be heated in 1°C increments on a programmable Linkam heating and freezing stage. All inclusions in a sample chip will be monitored following each 1° temperature increase, to determine whether or not the inclusion has homogenized. Thus, each chip will be subjected to a single heating run during which all inclusions will be monitored.

Following heating, inclusions will be frozen and ice melting temperatures will be evaluated to determine fluid salinities. Owing to the small size of the vapor bubbles, inclusions will probably require stretching (by overheating) so that a vapor bubble is present at the moment of ice melting, allowing an accurate determination of salinity.

A number of liquid-only inclusions, located in the older, intermediate, and younger parts of the crystals, will be evaluated by freezing to determine fluid salinities, and to identify any salinity variations that may be present. These inclusions will require stretching to generate a vapor bubble so that salinity can be determined.

Crushing studies have been performed in previous studies on vapor-only inclusions to determine the internal pressure exerted by inclusion gases. Dublyansky (1998b) reports crushing four vapor-only inclusions under oil, and observing shrinkage of the vapor bubbles. A photograph (Dublyansky, 1998b, see his Appendix 1, figure 3) shows minor shrinkage. He concludes shrinkage occurred as a result of inclusion trapping at elevated temperatures. Roedder and Whelan (unpublished manuscript) report that inclusions crushed under anhydrous glycerol exhibited no observable change in the volume of the vapor bubble, and they conclude that inclusions have an internal pressure of approximately one atmosphere. Whelan et al. (1998) suggest that Dublyansky's observations of bubble shrinkage may result from trapping of a fluid containing various gases plus some amount of water vapor. They suggest that cooling of the inclusion from 40° to 20°C would be sufficient for the water vapor to condense, resulting in the observed minor decrease in internal pressure. During the proposed study, we will crush vapor-only inclusions from old, intermediate, and young calcite, if available, under oil, and will monitor vapor bubble behavior. Results will provide qualitative constraints on the internal pressure of the inclusions, and will be evaluated in conjunction with compositional studies described below.

In addition, we will search for healed fractures and growth zones which contain coexisting liquid-rich and vapor inclusions that indicate fluid immiscibility. These inclusions would indicate the presence of coeval liquid and vapor.

Previous studies have attempted to identify the compositions of fluid inclusion gases and liquids, in an effort to identify inclusion populations, and to aid in constraining fluid sources. Qualitative quadrupole mass spectrometer analyses obtained by crushing small bulk samples showed that inclusions contain oxygen and nitrogen, in approximately atmospheric proportions, plus CO₂ and CH₄ (Roedder et al., 1994, Roedder and Whelan,

unpublished manuscript). Thus, Roedder and coworkers conclude that the oxygen and nitrogen reflect a system open to the surface; CO₂ and CH₄ may be of bacterial origin and may have exsolved from the aqueous fluid as it descended and the temperature rose slightly. Alternatively, gases could have been exsolved into the system from deeper circulating ground waters (Roedder et al., 1994; Roedder and Whelan, unpublished manuscript). Dublyansky evaluated vapor inclusion compositions using Raman spectroscopy, an undescribed selective absorption method, and bulk gas chromatographic analyses. Low intensity fluorescence of two vapor inclusions during Raman analyses indicated the minor presence of cyclic and aromatic hydrocarbons in the inclusions (Dublyansky, 1998b). Bulk chromatographic analyses indicated the presence of methane, acetylene, and hydrocarbons as heavy as C₆ (Dublyansky, 1998b). The semi-quantitative selective absorption analyses indicated the presence of 80 to 84 volume % methane and other hydrocarbons, 16 to 18 volume % nitrogen and noble gases, and trace carbon dioxide. Oxygen was not detected (Dublyansky et al., 1998b). Dublyansky (1998a) also reports the presence of liquid-only inclusions containing 11 wt.% MgCl₂; the analytical method was not indicated.

A number of nondestructive and destructive techniques for analyzing individual fluid inclusions to identify contained gases or dissolved components have been developed. These techniques include, among others, 1) ultraviolet fluorescence emission spectroscopy, to identify the presence and maturity of petroleum in inclusions, 2) synchrotron X-ray fluorescence (SXRF) to quantify or determine ratios of elements including S, Cl, K, Ca, higher mass alkali and alkaline earth elements and transition metals, 3) Laser Raman microprobe spectroscopy to identify and quantify polyatomic gases, 4) proton microprobe analyses (PIXE, proton-induced X-ray emission, and PIGE, proton-induced gamma ray emission) to quantitatively and qualitatively determine dissolved ions, 5) laser ablation, induced coupled plasma mass spectrometry (LA-ICPMS) analyses of single inclusions to quantify major and minor elements in the fluid, and 6) quadrupole mass spectrometry (QMS) analyses to quantify inclusion gases. The success of these techniques in providing information on inclusion composition depends on many factors including inclusion size and shape, depth to the inclusion, host mineral, fluid density, fluid composition, gas composition, and the presence of hydrocarbons. Owing to a lack of familiarity with the sample material and the contained fluid inclusions, I cannot currently assess which of these techniques might successfully provide compositional information. Funding is requested to investigate, during the course of the study, those techniques which appear to have a high probability of providing information on gas or solute composition. If successful, these analyses may aid in recognizing fluid inclusion populations, and contribute to constraining fluid sources.

The fluid inclusion study will require approximately four to six months. The time will be dependent on the number of fluid inclusion assemblages identified for analysis.

PHASE IV: Geochronology

- Tasks:**
1. Select, for geochronologic analysis, opal and calcite, which exhibit spatial relationships with two-phase fluid inclusions such that ages determined for these minerals can constrain the ages of the inclusions. Prioritize material for analysis based on 1) zoning, such that inclusions closest to the "young" end of the section are evaluated, and 2) inclusion origin, such that primary inclusions have the highest priority. This will lead to providing age constraints for the youngest inclusions, and providing both maximum and minimum age constraints, respectively.
 2. Collect sample material using a computer driven micro sampling device, which permits microscopic selection of sample locations and sample collection with a resolution of 10 micrometers.

3. Deliver sample material to laboratory for analysis.
4. Evaluate geochronologic data and determine age constraints for inclusions.
5. Evaluate SIMS isotope data in light of uranium series and U/Pb ages to further constrain ages of inclusion assemblages.

Procedures:

Detailed geochronologic studies (Paces et al., 1996; Paces et al., 1997; Neymark et al., 1998) have shown that small samples of calcite and opal (< 1 to 140 milligrams) from various regions within a calcite crystal, collected so as to minimize the number of growth zones analyzed, provide ages that substantiate progressive, incremental growth of calcite from the point of attachment to the tuff substrate, to the free growing crystal tips. Methods included radio carbon (^{14}C) dating of calcite, uranium-series ($^{230}\text{Th}/\text{U}$) analyses of opal and calcite on the outermost growth surfaces of samples, and uranium-series and U-Pb analyses of intermediate and old opal, and old chalcedony.

Uranium series ages determined for outermost growth faces (>100 analyses) range from 28 to 500 ka and exhibit a fairly even distribution of ages between 40 and 400 ka (Paces et al., 1996). Detailed studies of seven calcite specimens included U-Pb and uranium series analyses of three or four subsamples from each specimen including opal intergrowths in old, intermediate, and young calcite, and outermost growth surfaces, and chalcedony in old calcite (Paces et al., 1997; Neymark et al., 1998). Translucent gray chalcedony at the base of some of the samples provided the oldest ages of ~8 to 10 Ma (Neymark et al., 1998). Ages of opal from the older parts of the samples, located ~0.1 to 0.3 cm from the base of the specimens, range from 5.00 Ma to 7.67 Ma (Paces et al., 1997). Outer growth surface ages ranged from 94 to 407 ka. Samples from intermediate regions in all samples provided intermediate ages (Paces et al., 1997). These important results 1) document the protracted growth history of the calcite, 2) demonstrate the necessity of analyzing small samples containing a minimum number of growth zones, and 3) demonstrate that ages of fluid inclusion assemblages may be constrained by dating interior zones of opal or calcite that bracket the inclusion assemblages.

Samples will be selected for U-Pb and uranium series dating with the goal of identifying the youngest calcite that has trapped two-phase inclusions exhibiting consistent elevated homogenization temperatures. Fluid inclusion assemblages selected for dating will be prioritized based on 1) proximity to opal, owing to the smaller required sample size, as compared to calcite, 2) inclusion origin, with primary inclusions receiving a higher priority, and 3) their location within the sample, with inclusions located closer to the free growing surface of the sample receiving the highest priority.

Opal is the preferred mineral for dating owing to the smaller required sample size (~0.2 mg for U series and ~1 mg for U-Pb) (Paces et al., 1996; Paces et al., 1997). If opal is not available, calcite may be analyzed. However, because the concentration of uranium in calcite is several orders of magnitude lower than in opal (Paces et al., 1996), it is not possible to date calcite using U-Pb, and a significantly larger sample, approximately 40 to 140 milligrams, is required for uranium series dating. This larger sample may require analysis of material from more growth zones, resulting in a less accurate age. Also, only young calcite samples, < ~400,000 ka may be dated using this method. Absolute ages greater than ~400,000 ka cannot be determined, but calcites that exhibit secular equilibrium in uranium series isotopes are older than 500,000 years. Thus calcites that are younger than, and older than, ~500,000 ka can be distinguished.

In order to determine the age of fluid inclusions, it is critical to identify their origin. As primary inclusions are trapped during precipitation of the host mineral, ages of primary inclusions can be bracketed by obtaining ages for opal masses or calcite growth zones which have been determined, during the sample characterization study, to paragenetically bracket the fluid inclusion assemblage. Secondary inclusions are trapped at some unknown time after the formation of the host mineral. Maximum age constraints

may be placed on these inclusions by dating the paragenetically youngest calcite or opal cut by the fracture containing the fluid inclusion assemblage. While such ages will provide the youngest possible maximum age of the inclusion assemblage, a minimum age cannot be established.

As it has been demonstrated that calcite becomes progressively younger closer to the outermost sample surfaces, and as the last 500,000 years of geologic history of this region are of the greatest concern with respect to the proposed repository site, dating of the fluid inclusion assemblages will be prioritized such that assemblages located closest to the outermost growth surfaces have the highest priority. Thus, age constraints for the youngest assemblages of two-phase inclusions with consistent homogenization temperatures will be determined.

Geochronology results will be correlated with SIMS carbon and oxygen isotope analyses, to determine the degree to which isotopic ratios correlate with absolute ages. If a restricted relationship can be demonstrated, isotopic ratios will be determined for calcite growth zones bracketing evaluated fluid inclusion assemblages, further constraining the timing of fluid influx.

In order to provide a check on the SIMS results, a small number of conventional carbon and oxygen isotope analyses will be determined. Small, well characterized samples of individual, or a small number of growth zones will be collected using a computer controlled micro sampling device, which has a sampling resolution of 10 micrometers. The range of values determined for a single growth zone using SIMS, should fall within the range of values determined for the same growth zone using micro sampling and traditional isotope analytical methods.

The geochronology studies will take approximately four months, including analysis turn-around time.

PHASE V: Publication

Tasks: 1. Submit for publication in internationally-recognized, peer-review journals, manuscript(s) detailing the methodology and results of the study.

Procedures:

One or more manuscripts describing the petrographic, geochemical, fluid inclusion, and geochronological studies described above and all results, will be submitted for publication.

SUMMARY

This project will utilize 1) petrographic studies and geochemical analyses to thoroughly document the growth history of calcite samples, 2) fluid inclusion studies to determine salinities and homogenization temperatures of two-phase fluid inclusions in samples which have trapped a single-phase fluid with an elevated temperature, and 3) geochronologic studies to constrain the ages of these fluid inclusion assemblages. These results will indicate the presence or absence of influx, into the proposed repository site, of aqueous fluids with temperatures above current or recent ambient vadose-zone temperatures, and, if present, will constrain the timing of this influx. In addition this study will determine the areal extent of any identified fluid influx.

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PERSONNEL

1. **Principal Investigator - Jean Cline**, Associate Professor, University of Nevada, Las Vegas

The PI will be responsible for the successful and timely completion of the project under QA guidelines. The PI will oversee the studies that form the various

components of the project, which will be conducted by the post doctoral fellow (PD). The PI and PD will confer about data, will formulate and outline testing of proposed hypotheses, and will discuss interpretations and conclusions formed during the study. This will be accomplished through regularly-scheduled, daily meetings between the PI, PD, students, and other interested parties. The PI will oversee the writing and distribution of quarterly progress reports. The PI and PD will jointly manage quarterly meetings, during which time data and interpretations will be discussed with representatives from the State of Nevada and DOE, and an independent expert observer. The PI and PD will make presentations at an annual scientific meeting, and will disseminate final results through publication(s) in prestigious professional journals; the PD will be lead author on, and will take primary responsibility for, these communications.

The PI will expend three months per year on this project for two years.

The PI will be compensated by payment of three months salary, each year, for two years. This amount equals \$18,755 for the year beginning 7/1/99 (98-99 salary + 3%), and \$19,318 for the year beginning 7/1/00 (99-00 salary + 3%).

Total **\$38,073**

This compensation is consistent with other science researchers at the associate professor level at UNLV and other institutions.

2. Post Doctoral Fellow - search is currently underway

The PD will be responsible for conducting the research described previously. The PD will be responsible for sample collection, petrographic, and fluid inclusion studies. The PD will, furthermore, travel to Montrose, Colorado, to observe section preparation methods, and travel to selected laboratories to oversee or conduct the necessary geochemical analyses. The PD will confer with the PI concerning evaluation and interpretation of data and results. The PD and PI will jointly manage quarterly meetings with representatives from the State of Nevada and DOE, and an independent technical expert. The PD will write, with the assistance of the PI, quarterly progress reports. The PD will present results at an annual meeting, and will be lead author on publications resulting from the study. The PD will be responsible for detailing the study in a scientific notebook to fulfill QA requirements.

The PD will devote 100% of his or her time to this project for two years.

The PD will be compensated by a salary of \$35,000 to \$40,000 per year for two years.

Total **\$70,000-80,000**

This compensation is consistent with that paid to highly qualified geoscientists or geochemists with similar experience, at other institutions in the U.S.

3. Student Assistant I

A student will assist the PD with sample collection and preparation of samples prior to doubly polished section preparation. During sample collection the student will assist with physical removal and packaging of samples from the repository, note-taking to document the geologic context of the samples and fulfill DOE sample collection requirements, and transport of the samples from the repository. The

student will assist the PD in the selection and preparation of material for doubly polished sections. This work could be accomplished by the PD, but would extend the sample characterization study by two months.

This student will be hired, full-time, for a period of two months.

This student will be compensated at a rate of \$10 per hour.

Total

\$3360

This compensation is at the upper end of the wage scale paid to student workers. This wage will attract an intelligent and careful worker, and is justified, owing to the care that must be taken during sample collection and preparation. This student may be either a graduate student or upper-level undergraduate student.

4. Student Assistant II

The second student worker will assist the PI and PD with all aspects of the project including, but not limited to, xeroxing, filing, letter writing, sample collection (if additional samples are needed), petrography, photography, ordering, and preparation of figures for reports and publications. This student will be an undergraduate student, under close supervision by the PD, and will join the project at the termination of student assistant I. The purpose in hiring this student is two-fold: to release the PI and PD from some of the more mundane, but time consuming tasks associated with the project, and to acquaint a strong undergraduate student with the procedures and rigors of conducting careful research.

This student assistant will be a half time employee working ten months during the first year and twelve months during the second year.

This student will be paid \$10 per hour, twenty hours a week for 22 months.

Total

\$21,120

This compensation is at the higher end of the pay scale for student workers. This pay scale is necessary because it will allow us to attract and retain a highly qualified and motivated student, to work on a project that requires meticulous sample handling.

5. Electron microprobe technician.

An electron microprobe technician will be hired to assist with the set-up, calibration, and standardization of the microprobe. After the instrument becomes operational, the technician will be responsible for overseeing daily operation of the instrument and generation of accurate and precise data. The technician will provide expertise to facilitate collection of geochemical data and cathodoluminescence and surface imaging by the PD.

The technician will be employed full-time for a period of two years.

The technician will be paid a salary of \$40,000 plus benefits per year.

Total

\$80,000

This salary is comparable to salaries paid to other electron microprobe technicians in similar positions.

6. **Independent technical expert, Dr. Robert Bodnar, C.C. Garvin Professor of Geochemistry and Director of the Fluids Research Laboratory, Virginia Polytechnic Institute & State University.**

Dr. Robert Bodnar has been peripherally involved in the issue addressed by this proposal as an expert technical consultant for the Nuclear Waste Technical Review Board, and by providing laboratory facilities for recent studies by Yuri Dublyansky. Dr. Bodnar is a recognized expert in the study of fluid inclusions and he is acquainted with many of the individuals likely to participate in this study as representatives of DOE and the State of Nevada.

Dr. Bodnar will participate in the week-long, quarterly meetings with the PI, PD, and DOE and State of Nevada representatives, as an independent technical expert. His role will be that of facilitator (and referee) during discussions on issues such as sample collection, section preparation, analytical methods, petrographic interpretations, fluid inclusion analyses and interpretations, and chemical, isotopic, and geochronologic analyses. As a technical expert, not immediately involved in the study, but knowledgeable about the project, he will have a unique perspective that will allow him to facilitate group discussions, evaluate interpretations, referee scientific disagreements among group members, and promote compromise on issues such as methodology and data interpretation.

Dr. Bodnar will be paid a consulting fee of \$800/day, which will cover all expenses, for four, week-long meetings each year, for two years.

Total **\$32,000**

This rate was previously paid Dr. Bodnar as a consultant for the NWTRB.

TRAVEL

1. Travel to Yucca Mountain to collect samples. Four one-week trips will be made and the PD and student assistant will stay in Mercury during each week.

car rental from UNLV motor pool, \$6.75/day plus \$0.22/mile	20 days and 2000 miles	\$475
16 nights at Mercury x 2 persons, 32 nights at \$25/night		800
16 day's meals for 2 persons, 32 days at \$27/day		864
Total		\$2139

2. Four trips to Montrose, CO, to deliver samples and retrieve completed sections. During the first trip the PD will spend 2 days at the laboratory observing and documenting section preparation techniques. It is essential for the samples and sections to be hand carried to and from Colorado, to eliminate the possibility of their being subjected to unusually high or low temperatures, which could happen during transportation by mail, federal express, or in the baggage compartment of an airplane.

plane fare from LV to Montrose and return, 4 trips at \$435/trip (travel agent quote)	1740
car rental in Montrose, trip 1	150
cab fare to and from laboratory in Montrose, trips 2 to 4	100
1 night in Montrose (trip 1)	50
5 days meals	120
Total	\$2160

3. Two one-week trips to the University of Arizona in Tucson, to conduct LA-ICPMS analyses.

Plane fare LV to Tucson and return, 2 x \$150 (travel agent quote)	\$300
Car rental, 2 x \$250	500
Room and board, 8 nights at \$50/night for room, 10 days at \$24/day for food	640
Total	\$1440

4. Two one-week trips to UCLA, to conduct carbon and oxygen SIMS isotope analyses.

Plane fare LV to Los Angeles and return, 2 x \$150 (travel agent quote)	\$300
Car rental, 2 x \$300	600
Room and board, 8 nights at \$80/night for room, 10 days at \$24/day for food	880
Total	\$1780

5. Travel to Toronto to deliver samples and observe and document geochronology laboratory techniques.

Plane fare to Toronto and return, (travel agent quote)	\$400
Room and board, 2 nights at \$60/night, 2 days at \$27/day for food	174
Total	\$574

6. Travel to Reno, NV, for presentation at the Geological Society of America annual meeting, 11/2000.

Plane fare LV to Reno and return, 2 persons at \$78/person (travel agent quote)	\$156
Room and board, 4 nights per person at \$80/night for room, 4 days/ person at \$24/day for food, 2 persons	832
Transportation to and from airport in Reno, and local transportation, 2 persons	100
Total	\$1088

7. Travel from VPI&SU (R.J. Bodnar) to Las Vegas for quarterly meetings, and return, or, from Las Vegas to VPI&SU (PI and/or PD) for same.

Plane fare Roanoke, VA, to Las Vegas, or vice versa, and return, 8 roundtrips at \$500/trip (travel agent quote)	\$4000
Car rental, \$200/week	1600
Room and board, 16 nights per person at \$70/night for room, 16 days/ person at \$27/day for food	1552
Total	\$7152

ANALYSES

1. In-house electron microprobe costs to cover consumable supplies (liquid nitrogen, tape, scribes, cloth gloves, filaments, acetone, alcohol, and other miscellaneous supplies.) (Based on experience at the UNM probe lab, Rod Metcalf, 12/10/98)
\$80/sample for 150 samples \$12,000
2. LA-ICPMS geochemical analyses, 100 hours at \$70/hr 7000
(Quote from Joaquin Ruiz, University of Arizona, Tucson, 10/30/98)
QA calibration expenses (20% of analytical cost) 1400
3. SIMS carbon and oxygen isotope analyses, 10 days at \$1000/day 10,000
(Quote from Kevin McKeegan, UCLA, 12/15/98)
QA calibration expenses (20% of analytical cost) 2000
4. Fluid inclusion microthermometry and crushing expenses (liquid nitrogen, alcohol, acetone, oils for crushing studies, photographic paper) \$10/sample for 100 samples (Based on past experience in the UNLV fluid inclusion lab). 1000
5. Analyses of fluid inclusions to determine inclusion composition.

As described on pages 10 to 11 of the proposal, a number of nondestructive and destructive analytical techniques for analyzing individual fluid inclusions to identify contained gases or dissolved components have been developed. As we become familiar with the characteristics of the fluid inclusions in calcite during the course of the study, we will identify techniques which may provide information on inclusion compositions. This funding will support analytical costs to conduct these analyses. 20,000
6. U-Pb age determination with ²³⁴U-²³⁸U data, 15 analyses at \$1200

	per analysis (Quote from Yuri Amelin, Royal Ontario Museum, 11/16/98)	18,000
	QA calibration expenses (20% of analytical cost)	3600
7.	Combined U-Pb and ²³⁰ Th-U age determination, 15 analyses at \$1600 per analysis (Quote from Yuri Amelin, Royal Ontario Museum, 11/16/98)	24,000
	QA calibration expenses (20% of analytical cost)	4800
8.	Doubly polished section preparation, 300 sections at \$22/section. (1 section/sample for 200 samples, plus second section for 100 samples for cathodoluminescence and other destructive analyses) (Quote from Mark Mercer, Petrographic Services, 12/8/98)	6600
9.	Carbon and oxygen isotope analyses, 25 samples at \$60/sample (Quote from Chris Eastoe, University of Arizona, 12/22/98)	1500
	QA calibration expenses (20% of analytical cost)	300
	Total	\$112,200

EQUIPMENT

1. **Electron microprobe.** Purchase of an electron microprobe is included to allow us to conduct the integrated petrographic, micro geochemical, and cathodoluminescence analyses, and surface imaging studies required by this project. Critical to the successful completion of this study is the selection of appropriate material for fluid inclusion, geochemical, stable isotope, and geochronological analyses. Selecting this material can only be accomplished through detailed sample characterization studies which integrate petrography with electron microprobe chemical and cathodoluminescence analyses, and high resolution imaging. The petrographic and microprobe studies must be conducted, in tandem, so that petrographic features suggesting growth zoning, dissolution, or discontinuous growth, can be readily evaluated on the microprobe, and confirmed or rejected. For example, apparent growth zones, or discrete, but similar crystal morphologies identified under the microscope, would be examined on the microprobe for continuity of luminescing and nonluminescing band pairs, and chemical homogeneity. The capability to readily move back and forth between the petrographic microscope and the electron microprobe is necessary to produce the critically important paragenetic interpretations, which are essential in producing a high quality characterization study, and for the characterization study to move forward in an efficient and orderly fashion. The alternative option, traveling to use a microprobe at another laboratory, would eliminate the ability to readily move between the probe and the microscope, would require relying on another laboratory for a large amount of equipment time, which would be at their convenience rather than ours, and would be disruptive to identifying the temporal growth history of the samples. In summary, the ready availability of an electron microprobe will allow questions of stratigraphic continuity to be addressed and resolved quickly, will allow the study to be conducted in an efficient and timely fashion, and will greatly enhance the quality of the sample characterization study.

In addition, the electron microprobe is a fundamental piece of equipment, required for numerous geologic studies. If acquired, this equipment will be available for future studies at Yucca Mountain, or other sites of interest in Nevada.

Microprobe specifications. The instrument required for this project must be configured with three wavelength dispersive spectrometers and one energy dispersive spectrometer. This configuration will permit quantitative chemical analyses of major elements, and particularly trace elements at detection limits of 10 to 100 ppm, as required by this study. Back-scattered electron, secondary electron and cathodoluminescence detectors are also required to permit chemically distinct growth zones to be identified. The instrument also must have a goniometer stage assemblage to permit surface imaging of irregularly shaped samples, in addition to an X-Y-Z stage assembly for flat polished thin sections. The instrument must be available for delivery in late winter, so that analyses can begin in spring, 1999.

JOEL and Cameca microprobes. Two companies, JOEL and Cameca, manufacture electron microprobes; both instruments are suitable for this study and are described below.

A JEOL model JXA-8900R Automated Electron Microprobe is configured with three wavelength dispersive spectrometers (WDS) and one energy dispersive spectrometer (EDS) for quantitative chemical analyses of major and trace elements (<50-100 parts per million). The electron imaging system would include back-scattered electron (BSE), secondary electron (SE) and cathodoluminescence detectors. A color video camera system attached to a reflected and transmitted light polarizing microscope will allow petrographic examination of sample materials during analysis. Samples consisting of flat polished thin-sections will be handled using a high precision X-Y-Z sample stage. Irregularly-shaped samples for surface imaging will be handled using an interchangeable goniometer stage assembly. Fully automated control of the instrument will be handled by a host computer system (Hewlett-Packard B123L+) utilizing JEOL system software (sample stage drivers, spectrometers, imaging, and data reduction routines). As configured the instrument will allow integration of petrographic features, major and trace element microchemistry, and surface imaging.

JEOL JXA-8900R Automated Electron Microprobe with electron gun and optics, 3 WDS detectors, EDS, BSE SE detectors, geologic standards, automated sample stage, computer workstation, operational software, vacuum control system

Transmitted Light Illuminator	\$630,000
Optical Microscope Color Camera Adapter	\$24,500
Goniometer stage	\$6,250
Retractable Cathodoluminescence Detector	\$40,000
Air Cooled Recirculator	\$10,250
Inland Transport	\$4,960
Duty (if applicable)	\$2,500
Total	\$2,910
	\$721,370

The Cameca SX 100 Automated Electron Probe Micro-Analyser is equipped with three WDS detectors and one EDS detector for quantitative chemical analysis of major and trace elements. One of the WDS detectors would be outfitted with high intensity diffracting crystals which allows analysis of trace elements down to 10 parts per million. The electron imaging system would include BSE, SE and cathodoluminescence detectors. A color video camera system attached to a reflected and transmitted light polarizing microscope will allow petrographic examination of sample materials during analysis. The SX 100 is equipped with a stage shuttle system that allows quick interchange between a high resolution X-Y-Z platform for flat polished thin-sections and a rotation tilt goniometer platform for

irregular-shaped samples. Fully automated control of the instrument will be handled by a host computer system (SUN Desktop Ultra 1) utilizing Cameca system software (sample stage drivers, spectrometers, imaging, and data reduction routines). As configured the instrument will allow integration of petrographic features, major and trace element microchemistry, and surface imaging.

Cameca SX 100 Automated Electron Probe Micro-Analyser with electron gun and optics, 3 WDS detectors, EDS, BSE SE detectors, automated sample stage, computer workstation, operational software, vacuum control system, air cooled recirculator, optical imaging system	\$665,555
Geological Standards	\$8,680
System Software	\$5,680
Sample Holders	\$9,710
Video Monitor for Optical Images	\$1,320
Inland Transport	\$2,500
Duty (if applicable)	\$4,230
Total	\$697,675

Equipment availability. JEOL has two microprobes scheduled to come off the production line during the first couple months of 1999. Both of these instruments are available and could be delivered within thirty days of purchase. Cameca has a microprobe available for delivery in March, 1999.

During conversations with Joan Davis, Director, and Bob Ashby, Assistant Director of Purchasing at UNLV, we were assured that the bidding process for an electron microprobe could be completed within 30 days or less. We will include in our request for bids, that we be guaranteed instrument delivery within 30 days of completion of the bidding process. Thus, an instrument can be shipped to UNLV within 60 days of the starting date of this project.

Laboratory space. The UNLV geoscience department has identified a room to house this instrument, which is wired with the requisite power lines. Room renovation will require installation of a heating and cooling system to maintain room temperature, plumbing, and installation of laboratory cupboards and countertops. Renovation costs have been estimated by UNLV facilities personnel at \$30,000. The UNLV administration has committed to funding this renovation, and making renovation a top priority to be completed by the end of February, 1999. Thus, instrument delivery and set-up can be completed in time for analytical work to commence during spring of 1999.

2. **Micro sampling device.** Micromill is an X-Y-Z micro sampling device, designed for high resolution milling to recover sample powders for geochronological and isotopic analyses. Complex crystal growth zonation can be subsampled to provide a high resolution record of elemental and isotopic chemistries during fluid evolution.

The system provides a combination of submicron stage resolution and positional accuracy, real-time video observation, and a custom designed software system, that allows for sampling of complex crystalline materials. The system includes:

- High resolution, Y/C color CCD video camera for sample viewing and on-line digitization of image
- Motorized video

zoom 100X to 500X or 20X to 100X
 Fine resolution (.25 micron) automated XYZ stages with 2 or 3 inch travel
 Ring fiber optic illumination
 360-degree rotation for sample stage
 High precision, low eccentricity, high torque DC milling chuck with adjustable speed
 Carbide tipped or hardened cobalt steel milling tools for high resolution micro milling
 Pentium computer with 15 inch monitor
 Highest quality optics for best image quality and crisp sample viewing
 Optional joystick stage control for manual sample positioning

Micromill software features include:

Live video image displayed on computer monitor
 On screen digitization of image for micro milling
 Precise depth control over entire area of sampling (Z-axis tilt correction)
 Data record file with sample path information and estimated sample volumes
 Video frame acquisition and analysis

Micromill is the only instrument available which can provide these sampling capabilities, thus sole source purchase is justified. \$32,000

3. **Computer and laser printer.** Computer and laser printer for use by the PD. Price based on recent UNLV purchase. \$3500

4. **Digital camera for microscope.** A digital camera, which will be attached to an Olympus, research grade petrographic microscope, will be used to generate images to document and record 1) textural characteristics relevant to the sample characterization study, 2) fluid inclusion assemblages, and 3) opal and calcite growth zones sampled for traditional stable isotope and geochronological studies. The camera will also provide images for use as maps during the collection of geochemical and SIMS isotope data. 7000

MATERIALS AND SUPPLIES

Software for computer. A standard software package will accompany computer, but packages such as Adobe photoshop and Microcal Origin will be needed for manipulation of data, preparation of graphs and histograms, and generation of images from electron microprobe and other analyses.	1000
Miscellaneous supplies for the electron microprobe including sample holders, X-ray manuals, reference books, etc.	3000
Lane cabinet for sample storage	1400
Digital camera for field use to record sample collection sites and to provide a photographic record of samples collected	800
Black Light, to identify uranium-bearing opal during sample collection	225

Miscellaneous, xeroxing, mailing, telephone, pens	3000
Sampling materials: sample bags, markers, field books, small tape recorder and tapes, chisels	600
Total	\$10,025

RATE AGREEMENT
COLLEGES AND UNIVERSITIES

UNIVERSITY OF NEVADA, LAS VEGAS
4505 MARYLAND PARKWAY
LAS VEGAS, NV 89154

DATE: 01/30/97
FILING REF.: The preceding agreement was dated: 08/09/94 U50227.95

The rates approved in this agreement are for use on grants, contracts and other agreements with the Federal Government, subject to the conditions contained in Section II.

SECTION I : RATES

Type	Effective Period		Rate	Location	Applicable to
	From	To			
FACILITIES AND ADMINISTRATIVE RATES*					
Pred.	07/01/96	06/30/97	46.40%	ON-CAMPUS	SPONSORED RESEARCH
Pred.	07/01/97	06/30/99	49.00%	ON-CAMPUS	SPONSORED RESEARCH
Pred.	07/01/99	06/30/02	50.00%	ON-CAMPUS	SPONSORED RESEARCH
Pred.	07/01/96	06/30/02	26.00%	OFF-CAMPUS	SPONSORED RESEARCH
Pred.	07/01/96	06/30/02	59.70%	ON-CAMPUS	INSTRUCTION
Pred.	07/01/96	06/30/02	26.00%	OFF-CAMPUS	INSTRUCTION
Prov.	07/01/02	06/30/04		Same rates as cited above.	

*BASE: Modified total direct costs, consisting of all salaries and wages, fringe benefits, materials and supplies, services, travel, and subgrants and subcontracts up to the first \$25,000 of each subgrant or subcontract (regardless of the period covered by the subgrant or subcontract). Equipment, capital expenditures, charges for patient care and tuition remission, rental costs, scholarships, and fellowships as well as the portion of each subgrant and subcontract in excess of \$25,000 shall be excluded from modified total direct costs.

DEFINITION OF EQUIPMENT

Effective 07/01/96, equipment is defined as tangible non-expendable personal property having a useful life of more than one year and an acquisition cost of \$2000 or more.

TREATMENT OF PAID ABSENCES

Vacation, holiday, sick leave pay and other paid absences are included in salaries and wages and are charged to Federal projects as part of the normal charge for salaries and wages. Separate charges for the cost of these absences are not made.

TREATMENT OF OTHER FRINGE BENEFITS

This organization charges the actual cost of each fringe benefit direct to Federal projects. However, it uses a fringe benefit rate which is applied to salaries and wages in budgeting fringe benefit cost under project proposals. The following fringe benefits are treated as direct costs:

NEVADA INDUSTRIAL COMPENSATION, HEALTH INSURANCE, SUI AND RETIREMENT.

SECTION II: GENERAL

A. LIMITATION: The rates in this Agreement are subject to any statutory or administrative limitations and apply to a given grant, contract or other agreement only to the extent that funds are available. Acceptance of the rates is subject to the following conditions: (1) Only costs incurred by the organization were included in its facilities and administrative cost pools as finally accepted; such costs are legal obligations of the organization and are allowable under the governing cost principles; (2) The same costs that have been treated as facilities and administrative costs are not claimed as direct costs; (3) Similar types of costs have been accorded consistent accounting treatment; and (4) The information provided by the organization which was used to establish the rates is not later found to be materially incomplete or inaccurate.

B. ACCOUNTING CHANGES: If a fixed or predetermined rate is in this Agreement, it is based on the accounting system purported by the organization to be in effect during the Agreement period. Changes to the method of accounting for costs which affect the amount of reimbursement resulting from the use of this Agreement require prior approval of the authorized representative of the cognizant agency. Such changes include, but are not limited to, changes in the charging of a particular type of cost from facilities and administrative to direct. Failure to obtain approval may result in cost disallowances.

C. FIXED RATES: If a fixed rate is in this Agreement, it is based on an estimate of the costs for the period covered by the rate. When the actual costs for this period are determined, an adjustment will be made to a rate of a future year(s) to compensate for the difference between the costs used to establish the fixed rate and actual costs.

D. USE BY OTHER FEDERAL AGENCIES: The rates in this Agreement were approved in accordance with the authority in Office of Management and Budget Circular A-21 and should be applied to grants, contracts and other agreements covered by the appropriate regulation, subject to any limitations in A above. The organization may provide copies of this Agreement to other Federal Agencies to give them early notification of this Agreement.

BY THE ORGANIZATION

UNIVERSITY OF NEVADA LAS VEGAS
ORGANIZATION)

Norval F. Pohl
(Signature)

NORVAL F. POHL
(Name)

VICE PRESIDENT FOR FINANCE AND ADMINISTRATION
(Title)

2-11-97
(Date)

(CU)

BY THE COGNIZANT AGENCY
ON BEHALF OF THE FEDERAL GOVERNMENT
DEPARTMENT OF HEALTH AND HUMAN SERVICES
(Agency)

David S. Low
(Signature)

David S. Low
(Name)

Director, Division of Cost Allocation
(Title)

1/30/97
(Date)

HHS Representative Charles L. Travis
Telephone: (415) 437-7820

DE-FC08-98NV12081

Scientific and Engineering Studies of the Potential Yucca Mountain Repository

FLUID INCLUSION STUDIES

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OBJECT CLASS CATEGORIES:	10/23/98 to 6/30/99	7/1/99 to 6/30/99	7/1/00 to 6/30/01	7/1/01 to 6/30/02	7/1/02 to 6/30/03	TOTAL PROJECT
PERSONNEL:	\$110,213	\$111,610	\$0	\$0	\$0	\$221,823
FRINGE BENEFITS:	\$21,829	\$22,293	\$0	\$0	\$0	\$44,122
TRAVEL:	\$5,455	\$10,888	\$0	\$0	\$0	\$16,343
EQUIPMENT:	\$763,870	\$0	\$0	\$0	\$0	\$763,870
SUPPLIES:	\$3,310	\$6,715	\$0	\$0	\$0	\$10,025
CONTRACTUAL:(Consultant)	\$16,000	\$16,000	\$0	\$0	\$0	\$32,000
CONSTRUCTION:	\$0	\$0	\$0	\$0	\$0	\$0
OTHER:	\$36,075	\$76,125	\$0	\$0	\$0	\$112,200
TOTAL DIRECT CHARGES:	<u>\$956,752</u>	<u>\$243,631</u>	<u>\$0</u>	<u>\$0</u>	<u>\$0</u>	<u>\$1,200,384</u>
INDIRECT CHARGES:	\$94,512	\$121,816	\$0	\$0	\$0	\$216,327
TOTALS:	<u>\$1,051,264</u>	<u>\$365,447</u>	<u>\$0</u>	<u>\$0</u>	<u>\$0</u>	<u>\$1,416,712</u>
PROGRAM INCOME:	\$0	\$0	\$0	\$0	\$0	\$0

*** Staff will be added only if cooperative agreement approaches anticipated task and funding levels.
No additional fees will be charged for Administrative Services from Individual Tasks.

Patricia Baldwin
 PATRICIA BALDWIN, CRA - DIRECTOR of FINANCE
 MARJORIE BARRICK MUSEUM OF NATURAL HISTORY
 DATE: 1/6/99

PLEASE NOTE: Dependant upon the contingencies that may arise during research, the line items in the budget may be changed but the total amount will not be exceeded without further negotiation.

**D.O.E. COOPERATIVE AGREEMENT
DE-FC08-98NV12081
FLUID INCLUSION STUDIES
YEAR ONE**

PERSONNEL:	PERCENT TIME	HOURS	RATE (\$/hr) with Fringe Inc.	ROUNDED TOTAL
Principal Investigator: Jean S. Cline	25	450	\$50.43	\$18,755
Benefits @ 21%				\$3,939
Program Support				
Post Doc. Fellow	100	1800	\$29.24	\$43,498
Benefits @ 21%				\$9,134
Technician	100	1800	\$24.20	\$36,000
Benefits @ 21%				\$7,560
Student Technicians	50	1196	\$11.00	\$11,960
Benefits @ 10%				\$1,196
TOTAL HOURS:		5246	TOTAL SALARIES:	\$132,042
TRAVEL: Year One- 1/3 of total estimate SEE ATTACHED DETAIL (Includes Per Diem- Food and Hotel)				\$5,455
EQUIPMENT: SEE ATTACHED DETAIL				\$763,870
MATERIALS/SUPPLIES: Year One- 1/3 of total estimate SEE ATTACHED DETAIL				\$3,310
CONSULTANT: Dr. Robert Bodnar (Includes Travel Expenses)				\$16,000
OTHER:				
ANALYSES- Purchase Orders to Laboratories				\$29,075
CALIBRATION EXPENSES				\$3,000
SUPPLIES ASSOCIATED WITH ANALYSES, FED X CHARGES, DEMURRAGE				\$4,000
TOTAL DIRECT COST:				\$956,752
INDIRECT COST: .49 MTDC (DHHS Negotiated Rate)				\$94,512
TOTAL COST:				\$1,051,264

Hourly Basis is calculated
on 1800 hours/year.


PATRICIA BALDWIN, CRA-DIRECTOR of FINANCE
MARJORIE BARRICK MUSEUM OF NATURAL HISTORY
DATE: 1-6-99

PLEASE NOTE: Dependant upon the contingencies that may arise, the line items in the budget may be adjusted but the total amount will not be exceeded without further negotiation.

9/97

DRAFT DISCLAIMER

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