PROCEDURE: TEMPERATURE DETERMINATIONS FROM FLUID INCLUSION STUDIES

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1. PURPOSE

The purpose of this procedure is to describe methods of determining homogenization and freezing temperatures of fluid inclusions.

2. SCOPE

This procedure will be applicable whenever fluid inclusions must be examined in an effort to obtain data on past mineral-fluid interactions for the Yucca Mountain Project.

3. PRINCIPLES

Not Applicable.

4. DEFINITIONS

Not Applicable.

5. RESPONSIBILITIES

The principle investigator (PI) has the responsibility to assure implementation of this procedure for YMP-related fluid inclusion analyses. The PI may delegate performance of the procedure to any duly certified individual.

6. PROCEDURE

6.1 Overview

6.1.1 Equipment and Software Used

The equipment used for this procedure is an S.G.E., Inc. Fluid Inclusion Research System with a temperature range of -195.8°C (liquid nitrogen temperature) to 600°C. The practical range of temperatures for the YMP studies is from -150°C to 450°C. The S.G.E. system includes the following:

- An Olympus BH-2 petrographic microscope with x-y stage, adapted to use long-working-distance objectives with a Fluid Inc. substage condenser.
- A television camera with monitor (optional).
- A liquid nitrogen source (optional).
- A gaseous/liquid N_2 flow valving system.

- o A compressed air system for defrosting the heating-cooling stage window and for cooling the objective.
- o A rheostat with foot-pedal control for stage heating.
- o A trendicator-thermocouple (chromel-constantan type E) system for monitoring sample temperature.
- o No software is used.

6.1.2 Critical Laboratory Setup Parameters

None Applicable.

6.1.3 Special Environmental Conditions

None Applicable.

6.2 Sample Preparation

6.2.1 Chips of the rock used for standard thin section preparation are adequate. If epoxy impregnation is required, it is important that the sample be heated as little as possible, preferably not above 55°C; otherwise fluid inclusions that originally formed at lower temperatures may be ruptured.

6.2.2 The rock chip should be cut thick (about 1/2 centimeter thick), so that the final grinding and polish will leave a rock slice at least 100 μm thick, and preferably 200 μm thick, polished on both sides. The sample should be as thick as possible and still be transparent. Cutting, grinding, and polishing will be done in accordance with the Thin Section Preparation Procedure.

6.2.3 Chips may be mounted on glass slides with a cyanocrylate glue (superglue) rather than epoxy. This allows the chips to be removed from the slides by soaking in acetone. However, if the sample is going to be heated to temperatures greater than 80° C, the chips should be mounted with Crystalbond 509 or Lakeside 70 cement.

6.3 Preliminary Study and Final Preparation

6.3.1 Fluid inclusions are identified in the prepared sample by use of a petrographic microscope. Selection of appropriate fluid inclusions should follow the guidelines of E. Roedder (1984), Chapter 6. This reference also describes the step of sample photography for recording and relocating the inclusion for analysis. Photography is usually performed using a Polaroid system coupled to the analyst's petrographic microscope; other photographic systems are also be acceptable.

6.3.2 The thick section of the sample on a glass slide is placed in a shallow dish filled with acetone. After a time period that depends on a nature of the sample (a few hours to a day), the thick section will separate free from the glass slide.

6.4 Sample Analysis

6.4.1 The thick section is carefully cleaned with acetone and then broken into fragments that contain the photographed fluid inclusions. Fragments can be no larger than 2 cm across if they are to fit into the S.G.E. stage.

6.4.2 Insert the thick section fragment to be analyzed in the S.G.E. heating-cooling stage, following the instructions in Chapter III of the Fluid Inclusion System Manual (S.G.E., Inc.).

6.4.3.1 Cooling procedure (Chapter IV, S.G.E., Inc.) should be followed if fluid salinities are being sought and should always be conducted before heating if cooling is necessary. Methodology is described in Chapters 7, 8, and 9 of Roedder (1984).

6.4.3.2 Heating procedure (Chapter V, S.G.E., Inc.):
This is the procedure by which bubble homogenization temperatures are obtained and past temperatures of fluid-mineral interaction are determined. Homogenization temperatures of individual inclusions should be measured at least twice, and care must be taken to note whether a bubble immediately begins to grow upon cooling. If it does, the homogenization temperature has not been reached. In addition, great care must be taken not to heat the section so high as to break inclusions. Homogenization temperatures should be approached slowly and cautiously. The methodology to interpret the results of this analysis is described in Chapters 1, 2, 3, 4, 7, and 9 of Roedder (1984).

6.4.3.3 Modifications to the S.G.E., Inc. procedure:

A television camera has been mounted to the photographic column of the Olympus BH-2 used for fluid inclusion studies. This camera system has the following advantages:

(1) Magnification of the objective-ocular system on the Olympus is limited to 1000X. Using the television, this is boosted to 2060X. Fluid inclusions as small as 5 μm can be studied with the adapted television.
(2) Eyestrain is avoided by using the television rather than the microscope oculars.
(3) Temperature readings on the trendicator are easier to monitor because the analyst does not have to constantly look away from the oculars while tracking temperature.

6.5 Data Analysis

It is up to the individual investigator to determine in what form data shall be presented.

7. QUALITY ASSURANCE

7.1 Personnel

Sample selection, analysis, and data interpretation are performed by YMP-certified researchers with demonstrated working knowledge of the above-specified chapters of Roedder (1984) and the Fluid Inclusion System Manual (S.G.E., Inc.).

Demonstration that the operator has working knowledge of this procedure shall be his/her analysis of the Gonzales Fluorite Standard (Chapter XII, S.G.E., Inc.), as observed by the PI or QAL. The operator's analysis of the Gonzales Fluorite Standard will be documented in his/her controlled YMP notebook. Qualification will be documented in accordance with the YMP Personnel Certification Procedure.

7.2 Calibration

Calibration of the thermocouple and the trendicator should be completed yearly. Calibration will be accomplished in accordance with the Thermal Calibration Procedure (TWS-ESS-DP-105) or may be done by the Los Alamos Metrology Group and in accordance with the YMP Instrument Calibration Procedure.

7.3 Records

Documentation of sample identification, bubble homogenization temperatures on heating, bubble reappearance temperatures upon cooling, and freezing-point measurements (if applicable) will be recorded in the analyst's controlled YMP notebook. All photo documentation will be in the analyst's records. Notation that the sample has also been studied for fluid inclusions will be entered in the controlled YMP logbook maintained in the fluid inclusion analysis laboratory, as required by TWS-ESS-DP-101.

7.4 Accept/Reject Criteria

7.4.1 Homogenization or melting temperatures must be measured on a generation of inclusions to determine reproducibility. This process will always yield a range of temperatures. If significant variations in temperature are found (±30°C for homogenization and ±1 for melting), the measurements should be repeated to determine whether (a) the variations are real or (b) the variations are due to experimental errors. Significant outliers, e.g., T_H of 130°C when all other inclusions yield T_H from 230° - 260°C, should be rejected as due to an unsuitable inclusion and documented as such. Outliers may be the result of boiling or changes to the inclusion since it was trapped (e.g. necking).

7.4.2 Several potential sources of error are as follows:

- o Incorrect identification of inclusion origin.
- o Fluid immiscibility.
- o Not identifying all phases present, (e.g. CO_2)
- o Misinterpretation of boiling conditions. This leads to the single most common source of numerical error -- pressure corrections.
- o The dissolved solids may not be the only constituents contributing to the freezing point depression. Dissolved gases can contribute as well.
- o Cleavable minerals such as fluorite, barite, and calcite may give erroneous results due to inclusion stretching.

7.4.3 The entries in the analyst's YMP notebook and in the YMP Fluid Inclusion Logbook for a sample shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for that sample.

7.5 Procedural Deviations

Deviations from this procedure shall be fully documented in the analyst's YMP notebook explaining the deviation and the effects it may have on the resulting work.

7.6 Storage, Shipping, and Handling

Samples will be tracked, handled, shipped, and stored in accordance with the Procedure for Sample Identification and Control for Mineralogy-Petrology Studies (Ref 8.4). The equipment analytical requires no special storage, shipping, or handling considerations.

8. REFERENCES

8.1 E. Roedder (1984) Fluid Inclusions, Miner. Soc. Amer. Reviews in Mineralogy, Vol. 12, 644 pp.

8.2 S.G.E., Inc. (1983) Fluid Inclusion System Manual, TWS-ESS-1-4/85-4.

8.3 Sample Preparation Laboratory Procedures, Los Alamos National Laboratory TWS-ESS-DP-04.

8.4 Sample Identification and Control for Min-Pet Studies, Los Alamos National Laboratory TWS-ESS-DP-101.

8.5 Thermal Calibration Procedure, Los Alamos National Laboratory TWS-ESS-DP-105.

9. ATTACHMENTS

None.