USGS TECHNICAL PROCEDURE GCP-29, Rev 0

NNA.930816.0020

Thermoluminescence Dating

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1.0 PURPOSE.

1.1 To assure the accuracy, validity, and applicability of the methods used to determine ages of geologic samples using thermoluminescence (TL) dating; this procedure is a guide for U.S. Geological Survey (USGS) personnel and contractors performing the described activity.

1.2 This procedure describes the components of the work, the principles of the methods used, and their limits. It also describes the detailed methods to be used for calibration, operation, and performance verification of any equipment, if needed. In addition, it defines the requirements for data acceptance, documentation, and control; and it provides a means of data traceability.

2.0 SCOPE OF COMPLIANCE.

2.1 This procedure applies to all YMP-USGS personnel and their contractors who may perform work referred to in Para. 1.1, or use data obtained from this procedure.

2.2 For all technical activities, data collected from using this procedure and any equipment calibrations or recalibrations that may be required shall be in accordance with this technical procedure. Variations are allowed only if and when this procedure is formally revised, or otherwise modified, as described in Para. 8.

3.0 PERSONNEL RESPONSIBILITIES. The Principal Investigator (PI) is responsible for assuring full compliance with this procedure. The PI shall require that all personnel assigned to work to this procedure shall have the necessary qualifications and training to adequately perform the procedure; and they shall have a working knowledge of the YMP-USGS QA Program. Responsibilities of others including the reviewer(s); contributing investigators; Chief, Geologic Studies Program (GSP)/Chief, Hydrologic Investigation Program (HIP); QA Office; and the Chief, Yucca Mountain Project Branch (YMPB) are as described in Para. 4, YMP-USGS-QMP-5.01. When procedure-specific responsibilities are to be delegated to contributing investigators or other personnel, the details of these responsibilities are as stated in this procedure. Special qualifications and/or training unique to the conduct of this procedure are as follows: a background in physical science. As necessary, all ongoing investigations shall be identified, at the location of the scientific investigation, to preclude inadvertent interruption and to ensure compatibility of the investigations.

4.0 DETAILED PROCEDURE. The principles of TL dating are described by Aitken (1985) and Forman (1989). Radiation causes electrons to be caught up in 'electron traps' in the crystal lattice of the mineral grains. The equivalent dose (ED) is a measure of accumulated radiation exposure due to background radiation from

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the radioactive decay of ^{238}U, ^{232}Th, and ^{40}K, and bombardment by cosmic rays. Ages are determined by dividing the equivalent dose by the dose rate (DR) which is an estimate of the background radiation.

4.1 Objective: The objective of this study is to obtain numerical ages for sediments using TL techniques.

4.2 Methods Used: The principles of TL dating are described by Aitken (1985) and Forman (1989). The procedures currently in use for dating of sediments are summarized by, but not limited to Aitken (1985) and Singhvi (1985).

4.2.1 SAMPLE COLLECTION: Samples collected under this procedure will be recorded on a TL Sample Description Sheet (Attachment 1). Samples will be collected in the following manner: The site of sample collection should be as free as possible of roots, carbonates, and stones. An approximately 1-foot-deep hole must be augured into the soil and a sample tube driven into the back of the hole to collect the TL sample. Using a black, opaque cloth to shield the sample from light, the sample tube is carefully removed from the hole and the open end closed with a black cap. Both ends of the tube should be sealed with black electrical tape and the tube placed in a black polyethylene bag. A second sample must be collected from the back of the same hole. This sample, referred to as the 'bulk' sample, does not need to be protected from light. The bulk sample is collected in a clear Ziploc bag that inhibits the loss of moisture from the sample, so that moisture content can be measured back in the laboratory.

4.2.2 *IN SITU* GAMMA COUNTING: In order to obtain gamma spectra, the sample hole must be deepened to a depth of approximately 2 feet. The NaI gamma detector (Exploranium Portable Gamma Spectrometer) is inserted into the enlarged hole and two 999-second count spectra are collected to determine the dose rate. See the Terraplus manual for spectrometer operating instructions.

4.2.3 BULK SAMPLE AND MOISTURE CONTENT: The bulk sample is used to determine moisture content in order to better model the environment in which the sample was collected. Moisture absorbs beta particles and some gamma radiation thus reducing the radiation dose to the sample. Weigh the whole sample using a laboratory balance. Allow the sample to air dry for several days. Reweigh the sample and compute moisture content.

4.2.4 TL SAMPLE PREPARATION: In a darkened room, open sample tube and remove approximately 1" of material from each end of the tube and discard. Section the remaining sediment into thirds. Place 2/3 (approximately 200 g) of the material into a 1/2 pint paint can, seal, and store as a reserve split. Place the remaining 1/3 (approximately 100 g) of material into a 600-ml beaker and store in dark cabinet until pretreatment.

4.2.5 TL SAMPLE PRETREATMENT: This procedure must be carried out in a darkened room. Pretreatment involves treating the sediment with various solutions. Solutions are added one at a time to the material in the 600-ml beaker. After each solution is added, the material is allowed to settle and the supernatant removed with an aspirator. While settling, the

sample must be kept in a dark storage cabinet. The following solutions are used in sequence:

4N HCl – enough to wet the sediment
Deionized water – 500 ml (x2)
30% H_2O_2 – 100 ml (x2)
Deionized water - 500 ml
50 g/L Na pyrophosphate – 50 ml (sonicate for 10 min.)

4.2.6 SILT SEPARATION: The following procedures are used to separate the 4-11 μm silt used for TL dating.

4.2.6.1 Wet Sieving: This procedure must be conducted in a dark-room. Wash the pretreated sediment through a 63 μm sieve with approximately 900 ml of deionized water. Catch the P63 μm sediment (P=pass) in a bucket and transfer to a 1000-ml beaker. Wash the R63 μm sediment (R=retained) into a 250-ml beaker with methanol. Store both in a dark cabinet.

4.2.6.2 Stoke's Settling: This procedure may be carried out under sodium vapor lights. Transfer the P63 μm slurry to a 1000-ml plastic graduated cylinder and add deionized water to bring up to 1000 ml. Shake or stir the slurry and let stand for 1 hour in a dark storage cabinet. Decant the <11 μm suspension into a 1000-ml beaker and discard the 11-63 μm sediment.

4.2.6.3 Centrifugation: Under the sodium vapor lights, pour 30 ml of the <11 μm suspension into each of four 40-ml centrifuge tubes. Using the laws of Stoke's Settling, centrifuge, and aspirate to remove the <4 μm particles. Repeat this procedure until there are approximately 100 mg of sediment in the bottom of each tube. Wash the 4-11 μm sediment three times with approximately 15 ml of methanol, centrifuging and aspirating after each wash. Add sediment from two centrifuge tubes to a 4- or 8- oz. bottle and fill with methanol to 100 ml. Pipette approximately 1 ml of suspension onto a tared counting planchet, dry, and weigh to determine mg/ml. Add more sediment or decant methanol until suspension is 1.5-2.0 mg/ml. Prepare a second 100 ml in the same manner. Allow the remaining <11 μm suspension to settle overnight. Transfer the sediment to a 150-ml beaker with deionized water and allow to stand for one hour. Aspirate, and transfer the sediment to tared 35 mm film canister and record weight. Seal the film canister with black electrical tape, and store as a reserve split.

4.2.7 DISK PREPARATION AND NORMALIZATION: Place approximately 100 1-cm Al disks into flat-bottomed shell vials. Pipette approximately 1 ml of the 4-11 μm suspension into each vial. Allow the methanol to evaporate in a laboratory oven. Transfer the disks to black, opaque, plastic cell well boxes.

Normalization of the disks is carried out by Infrared Stimulated Luminescence (IRSL) using the TL instrument. This involves exposing each disk to infrared light for the same amount of time. That time does not need to exceed 5 seconds. This allows some trapped electrons to be released, giving off a luminescence signal. The amount of sediment, and the even-

ness of deposition varies from disk to disk. Therefore, it is necessary to normalize all of the disks to one disk. After normalization, return all of the disks to the cell well boxes, taking care to keep the disks in the same order in which they were normalized.

4.2.8 TL Experiments: Four TL experiments are carried out under this procedure. They include a sunlight bleach experiment, a total bleach experiment, a partial bleach experiment, and an anomalous fading experiment. It is not necessary to carry out all four experiments, although it does help to refine age estimates, but at least the total bleach and partial bleach experiments must be conducted. Ideally, in each experiment, 4 disks make up each data point; fewer disks are acceptable but at least 2 should be used.

1) Sunlight Bleach: This experiment is used to validate the sunlight bleaching times for the total and partial bleach experiments. Set aside 4 natural disks (disks that have been normalized only). Eight sets of 4 disks are then exposed to full sunlight, each set for a different amount of time. The following time periods are used: 5 min., 10 min., 30 min., 1 hr., 2 hrs., 6 hrs., 16 hrs., and 32 hrs. After bleaching, glow all of the disks (see Para. 4.2.9).

2) Total Bleach: This is used to determine the total bleach ED of the sample. Set aside 4 natural disks, bleach 4 disks in sunlight for 16 hrs., and irradiate a set of 4 disks for each of 4-5 dose data points. The dose data points are appropriate multiples of the ED, where the estimated ED is equal to the natural dose rate of the sample, multiplied by the estimated age of the sample (ED = DR x est. age). Preheat all of the disks on a hot plate and glow the disks (see Para. 4.2.9).

3) Partial Bleach: The partial bleach ED of the sample is determined combining data from this experiment with data from the total bleach experiment. Set aside 4 natural disks. Irradiate sets of 4 disks for the same dose data points as used in the total bleach experiment. Bleach all of the disks for the same amount of time. This time period should be less than an hour. Preheat and glow all of the disks. Plot the TL signals against the doses of both the total and partial bleach experiments. The partial bleach ED is determined by the intersection of the two lines.

4) Anomalous Fading: Preheating, as used in the total and partial bleach experiments, removes the least stable TL component. To determine how effective the preheating is, irradiate 8 disks for about 2.5 hours. Glow 2 of the disks immediately after irradiation, and store 2 of the disks for several weeks before glowing. Preheat the remaining 4 disks, glowing 2 of them immediately after heating, and storing the other two for several weeks before glowing.

For further discussion of these TL experiments, refer to Aitken (1985) and Singhvi (1985).

4.2.9 GLOWING: In the TL instrument, each disk is heated to 500°C such that the temperature increases at a rate of 5°C/sec. The TL signal,

consisting of photon counts emitted from the sample, is collected at 5°C increments. The glow curve is a plot of the intensity of the photon counts against temperature.

4.2.10 OPERATIONAL CHECKS: To ensure the TL instrument and the beta irradiator are functioning properly, operational checks are carried out by the investigator by comparing results from materials analyzed both in the Denver Federal Center TL lab and other TL labs. Results should agree to within about 10%. Operational checks shall be carried out when TL data is nonconforming with the requirements of Para. 4.5.1.

4.3 Materials/Equipment Required: All materials and equipment listed below shall be as per listed manufacturer or equivalent.

4.3.1 SAMPLE COLLECTION:

- o Exploranium portable gamma spectrometer with 3" x 3" NaI gamma detector (5% precision)
- o Sample collection tubes (6" piping) and black caps
- o Black, opaque polyethylene bags
- o Ziploc 1-quart freezer bags
- o Black electrical tape
- o 4-inch auger with 2-foot extension rod and handle
- o Drive tube and sledge hammer
- o Black, opaque cloth

4.3.2 SAMPLE PREPARATION AND PRETREATMENT:

- o Dark storage cabinet
- o Triple-beam laboratory balance - approximately ± 0.1 g accuracy; range 0 - 210 g (For indication only)
- o 150, 250, 600, 1000-ml beakers
- o Aspirator
- o 63 μm sieves
- o 1000-ml plastic graduated cylinders
- o Centrifuge
- o 40-ml centrifuge tubes
- o 4-, 8- oz. glass bottles
- o Darkroom
- o Sodium vapor lights
- o Laboratory reagents (commercial grade):
  - Deionized water
  - 4N HCl
  - 30% H_2O_2
  - 50 g/L Na pyrophosphate dispersant
  - Methanol
- o 1-cm diameter Al disks
- o Glass shell vials
- o 6" x 4" black, opaque, cell well boxes
- o Laboratory oven
- o 35 mm film canisters
- o 1/2 pint paint cans
- o Pipette
- o Counting planchets
- o Bucket

- o Electrical tape

4.3.3 NORMALIZATION, IRRADIATION AND GLOWING FOR TL EXPERIMENTS

- o Hot plate for preheating disks
- o Daybreak TL instrument (see Bortelot, 1991 for range and accuracies)
- o IRSL adapter for TL instrument (see Bortelot, 1991 for range and accuracies)
- o Optical filters (refer to Aitken, 1985, for discussion on filters)
- o Daybreak beta irradiator (2.79 grays/min. ± .05)

4.4 Assumptions Affecting the Procedure: For this procedure it is necessary to assume that sample material has been sufficiently exposed to sunlight during geologic transport, thus bleaching the material and resetting the geologic clock.

4.5 Data Information: Sample collection sheets shall record, at a minimum, the information required of YMP sample collection reports per AP-6.26Q. Preparation of both the TL and bulk samples is recorded on lab worksheets. These worksheets record, at a minimum, sample identification, date begun and date completed, and identification of the investigator. Completed analytical data sets obtained from the Daybreak TL instrument of TL signals will be printed out in numerical and graph form with backup on hard disk, and will record sample ID, date of analysis, and identification of the investigator.

4.5.1 QUANTITATIVE/QUALITATIVE CRITERIA: Ideally, each data point in these experiments consists of data from a set of 4 disks. During disk preparation (Para. 4.2.7), disks may be unevenly covered with sediment. It is up to the investigator to determine if there is enough sediment evenly spread on the disk. Disks that produce odd shaped glow curves, or curves that deviated from the others in its group by more that 3 sigma, should be discarded. Ultimately, a plot of the ED and temperature should result in a curve with a plateau. It is up to the investigator to determine if the parameters of the plateau are acceptable. See Berger (1987, 1988a) for more on plateau tests. Measurements of volume, temperature, radiation, and particle size are approximate in nature and absolute determination of these parameters are not necessary for successful performance of the analysis.

4.6 Limitations: The application of TL dating is best applied to materials less than 120 ka. There is much debate about the farthest limits of the application. Certain materials can be more accurately dated using certain experiments. Age limitations and material constraints are discussed by Berger (1992), Forman (1989, 1991), and Singhvi (1985).

4.7 Other: The TL instrument is run using 1100SI Applications software for Daybreak 1100 automated TL System (Bortelot, 1991).

5.0 CALIBRATION REQUIREMENTS. Calibration is required as a part of this technical procedure. All instruments and/or instrument systems shall be calibrated in compliance with the YMP-USGS-QMP-12.01, Instrument Calibration for producing data under graded QA controls.

5.1 Calibration Responsibility: The PI is responsible for calibrations required by this procedure. Calibration shall be in accordance with procedures described or referenced in Para. 5.2. Maintenance of all calibration records described in Para. 5.3 may be done by a contributing investigator under the direct supervision of the PI.

5.2 Calibration Procedures: All calibrations, unless otherwise specified, shall be performed according to manufacturer's range and accuracy specifications.

5.2.1 EXPLORANIUM PORTABLE GAMMA SPECTROMETER: The portable gamma spectrometer is calibrated independently of the laboratory by performing counts on the calibrated radiation pads at the DOE Field Calibration Facilities for Environmental Measurement of Radium, Thorium, and Potassium, in Grand Junction, CO (Steele, 1986). Readings should vary by no more than 15%. See the Terraplus instruction manual for procedures on using and testing the spectrometer. This calibration shall be carried out by the investigator at least once every two years.

5.3 Calibration Records: In compliance with YMP-USGS-QMP-12.01, calibration data will be entered, signed and dated, into a notebook or other organized documentation. Notebooks and other documents shall be protected and submitted to the LRC in accordance with YMP-USGS-QMP-17.01 (YMP-USGS Records Management).

5.4 Labeling of Equipment Calibration Status: Labeling of equipment shall be in compliance with YMP-USGS-QMP-12.01.

6.0 IDENTIFICATION AND CONTROL OF SAMPLES. Samples will be collected and handled in accordance with Para. 4.2.1 of this technical procedure.

6.1 Sample Identification: As part of the data records and documentation and in compliance with YMP-USGS-QMP-8.01, all samples shall be identified as follows: A unique sample number, TL - sequential digit, will be assigned by the investigator in the TL laboratory. Sample identification shall also meet the requirements of Department of Energy administrative procedure AP-6.26Q, Submission and Documentation of Non-Borehole Samples to the Sample Management Facility.

6.2 Control and Storage: In compliance with YMP-USGS-QMP-8.01, the collected and identified samples shall reside in the custody of the investigator who shall store them in a secured area until used in analysis.

6.3 Special Treatment: Samples may only be handled in a darkened room or under sodium vapor lights. as indicated within these procedures. When not being prepared or analyzed, samples must be stored in a dark storage cabinet.

7.0 QUALITY ASSURANCE RECORDS. Documents and data will be prepared and submitted per appropriate governing project procedures.

7.1 Anticipated documents and data generated from implementation of this procedure may include the following: sample collection sheets, lab work-

sheets, and printouts of TL signal data. Copies of these records will be submitted to the Local Records Center.

7.2 Notebooks, forms, or other organized documentation shall be prepared, as appropriate, by the PI or a contributing investigator to record data from this procedure and shall include the date of data collection, the identification of the investigator collecting the data, the unique identifier of equipment used to collect/record the data, and any information considered by the originator to be pertinent. When in loose-leaf form, each page shall be numbered consecutively and chronologically. Any revisions shall be lined out, initialed, and dated. Notations by pencil shall be submitted in legible photocopy form.

8.0 MODIFICATIONS. When modifications or expedited changes become necessary, the PI shall fully document the changes in compliance with YMP-USGS-QMP-5.01 and submit the documentation for review, approval, and distribution as indicated.

9.0 REFERENCES CITED.

Aitken, M.J., 1985, Thermoluminescence Dating: Academic Press, NY.

Berger, G.W., 1987, Thermoluminescence dating of the Pleistocene Old Crow tephra and adjacent loess, near Fairbanks, Alaska: Canadian Journal of Earth Sciences, v. 24, p. 1975-1984.

Berger, G.W., 1988a, Dating Quaternary events by luminescence, in Easterbrook, D.J., ed., Dating Quaternary sediments: Geological Society of America Special Paper 227, p. 13-50.

Berger, G.W., Pillans, B.J., and Palmer, A.S., 1992, Dating loess up to 800 ka by thermoluminescence: Geology, v. 20, p. 404-406.

Bortelot, V., 1991, 1100SI TL Applications Software for Daybreak 1100 Automated TL System, ver. 3.23, Daybreak Nuclear and Medical Systems, Inc., Guilford, CT.

Forman, S.L., 1989, Thermoluminescence Dating: Dating methods applicable to Quaternary geologic studies in the western United States, p. 24-36.

Forman, S.L., Nelson, A.R., McCalpin, J.P., 1991, Thermoluminescence dating of fault-scarp-derived colluvium: deciphering the timing of paleoearthquakes on the Weber segment of the Wasatch Fault Zone, north central Utah: Journal of Geophysical Research, v. 96, p. 595-605.

Singhvi, A.K., and Mejdahl, V., 1985, Thermoluminescence dating of sediments: Nuclear Tracks, v. 10, p. 137-161.

Steele, D.W., and George, D.C., 1986, Field calibration facilities for environmental measurement of radium, thorium, and potassium: U.S. Department of Energy.

Terraplus USA, Inc., Portable gamma ray spectrometer, model GR-256 with model GPS-21 detector: Littleton, CO, 62 p.

10.0 ATTACHMENTS.

Attachment 1: Sample Description Sheet

11.0 SUPERSEDED DOCUMENTS. None.

12.0 APPROVALS AND EFFECTIVE DATE.

EFFECTIVE DATE: 7/26/93

Z.E. Peterman 7/9/93
PI: Z.E. Peterman Date

for Martha H Mustard 7-20-93
YMP-USGS QA Manager Date

Paula Maat 7/13/93
Tech. Reviewer: Paula Maat Date

7/19/93
Chief, YMPB Date

for Bruce Thurber 7/20/93
Chief, GSP: J.S. Stuckless Date

USGS, Box 25046 MS 963
Denver Federal Center
Denver, CO 80225
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YMP-USGS THERMOLUMINESCENCE PROJECT
Sample Description Sheet

Project Sample Number: TL- _____

Field Sample Number _____ Date _____ Collector _____

Locality Description:

Site Name: _____

Elevation: _____ ft.

Latitude: _____

Longitude: _____ "attach bar code label here"

Quadrangle: _____

Other Reference Point(s):

_____

Spectrometer ID Number: _____

Battery Check: _____

Time	Geom	CT	TC	K	U	Th	CR	Spec #

Sketch

Type of Sample: ☐ Gravel ☐ Sand ☐ Vesicular A ☐ Soil ☐ Other _____

Pictures

_____

_____

_____

_____

Comments

_____

_____

_____

_____

Special storage requirements? ☐ N ☐ Y _____

Released to SMF? ☐ N ☐ Y _____ (initials)

REVIEWER SELECTION FORM

[Part A and/or Part B to be completed by the Chief, Geologic Studies Program/ Hydrologic Investigations Program, Chief, Yucca Mountain Project Branch or Quality Assurance Manager responsible for reviewer selection.]

Document(s) to be reviewed:

NWM-USGS-GCP-29, R0 - Thermoluminescence Dating

Name of reviewer: Paula Mart

Title of reviewer: Geologist

PART A:

Selection of reviewer not drawn from YMP-USGS:

Employer of reviewer: USGS, Branch of Isotope Geology

Basis of selection for reviewer: This reviewer has been selected on the basis of her work experience and education and knowledge of the techniques of thermoluminescence dating

John S. Stuckless — Chief, Geologic Studies Program
Printed Name — Title

John S. Stuckless — 6/4/93
Signature — Date

PART B:

Although this reviewer is or has been indirectly involved in the development of the document, he/she is the only qualified person available.

N/A
Printed Name — Title

N/A
Signature — Date

N/A
QA Manager's Approval — Date