Best practices for collection of luminescence samples and estimating water content for dose-rate determination: Why sample selection, collection technique and water content matters

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Luminescence dating provides an age estimate of the last time quartz or feldspar minerals were last exposed to sufficient light or heat. The signal (photons of light) is generated after an electron recombines in a lower energy state following stimulation.

$$\text{Luminescence Age (ka)} = \frac{\text{Equivalent Dose (Gy)}}{\text{Dose Rate (Gy*ka}^{-1})}$$

Optically stimulated luminescence (OSL) - utilizes quartz for dating

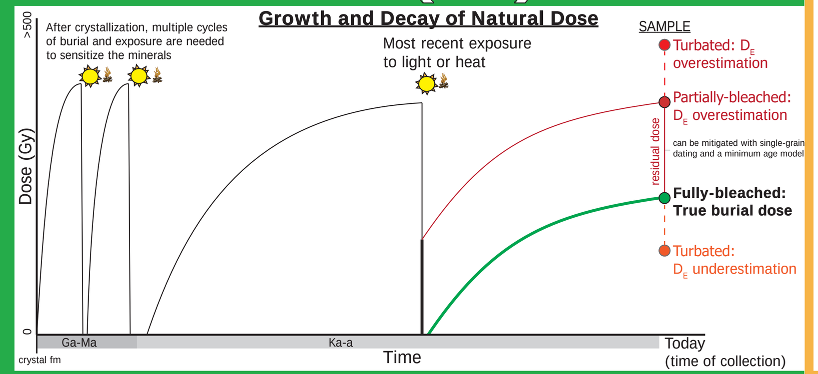
Infrared stimulated luminescence (IRSL) - utilizes potassium feldspar

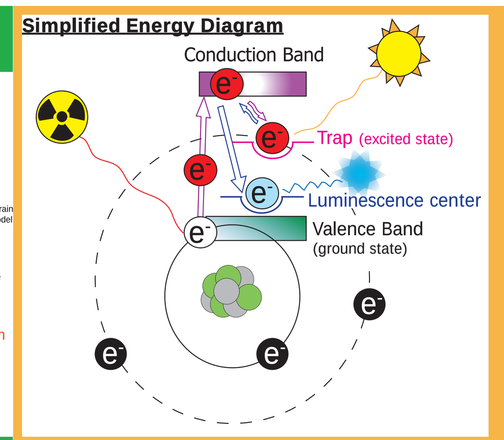
Thermoluminescence (TL) - uses heat to measure the equivalent dose

Equivalent Dose (D_E)

The amount of absorbed radiation since last exposure to light or heat.

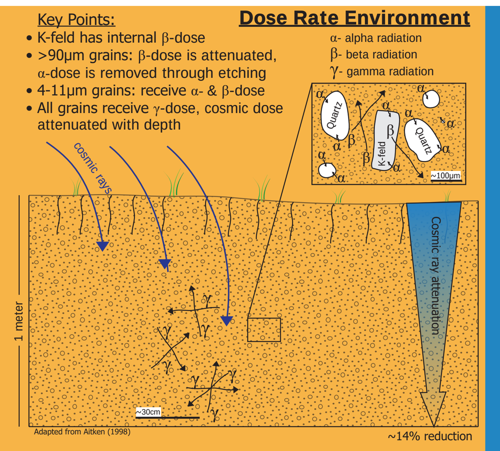
For dating, this sample must not be exposed to light.





Dose Rate (D_R)

Rate in which trapped electrons accumulate in traps, and is proportional to the flux of radiation from radioelemental decay of potassium, uranium, thorium, and rubidium, in addition to cosmogenic nuclide radiation



Water Content

Sediment pore-space water content absorbs more radiation per unit mass than air-filled pore spaces. Environmental D_R is diluted as sub-atomic particles produced by nuclear decay are attenuated by the presence of water molecules.

We present a new approach for mitigating uncertainties associated with non-representative in-situ water content measurements, calculation of F (pore-space occupied by water), and measurement of saturation values by utilizing a grain-size-based model and the van Genuchten (1980) water retention equation.

Equation for attenuation and D_R conversion on radiogenic particle concentration:

$$\frac{[R]*b*c}{1+x*W*F}$$

[R]= concentration of U, Th, K, Rb
b= alpha and/or beta attenuation coefficient
c= conversion factor
W= saturated gravimetric water content **(rarely known, can be estimated)**
F= fraction of pore-space occupied by water **(subject to sampling conditions)**
x= water attenuation factor

van Genuchten (1980) water retention curve (WRC) equation :

$$\theta(h) = \theta_r + \frac{\theta_s - \theta_r}{[1+(\alpha|h|)^n]^{1-1/n}}$$

θ_s: saturation water content, cm³/cm³
θ_r: residual water content, cm³/cm³
h: matric potential, -kPa or -cm H_2O
α (1/cm) & n: curve shape parameters

- Grain size requirements: 90-250µm or 4-11µm
- Target quartz (SiO_2) or potassium feldspar ($KAlSi_3O_8$)
- Avoid stratigraphy with indicators of bio-, cryo-, pedoturbation
- Requires sufficient exposure to light or heat to reset any previous (unwanted) signal
- Some geologic source areas may produce weak luminescence signals- low sensitivity
- Dating range is typically ~100 - 200,000 years, or greater depending on dose rate environment

- Requires representative sample collection of surrounding stratigraphy
- Geographic location information is important for calculation of cosmogenic D_R
- Dose rate attenuated by water content and non-linear with variability
- Determined through chemical analysis, in-situ or laboratory radiation detection
- Chemical and physical weathering can remove or add radioelements in sediment

- Dose rates used in age calculation for luminescence and other radiometric dating techniques must account for the amount and variability in water content, over or underestimation of this factor will lead to inaccurate age results
- In-situ moisture content measurements can suffer from non-representative conditions at the time of sampling or effects of surface drying at the exposure front
- It is often difficult to replicate the level of compaction of the sediment in the field which is needed to calculate saturated water content
- Pedotransfer functions (PTFs) are frequently used in soil science surveys to translate field data into predictive soil hydraulic information (e.g. water content and hydraulic conductivity), which are often difficult to measure in-situ due to the time and instrumentation required

1) Contact the luminescence laboratory prior to sample collection
2) Sample collection methods and materials

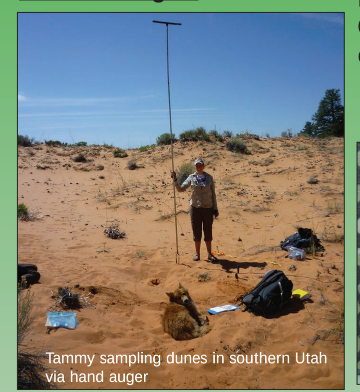
Outcrop/Exposure - Tube method:


Michelle sampling alluvial sediments near Kanab, UT

- Clean off exposure
- Target fine-medium sand with original bedding
- Avoid soils, bio-, or cryoturbated sediments
- Sample a thick unit with relatively uniform sediment
- Use pounding cap to drive STEEL tube into sediment, fill completely with sediment to minimize mixing during transport to lab; **avoid using PVC**

Subsurface- Core method:

Manual- auger:


Tammy sampling dunes in southern Utah via hand auger

Pros: inexpensive, packable
Cons: limited depth, difficult in dense sediments

Types of auger tubes and bits:

Geoprobe or Giddings hydraulic auger:
Pros: in-situ and deeper collection
Cons: expense, portability/availability


Geoprobe sampling in Alaskan basin sediments
Credit- Tim Nelson, Univ. New Orleans

Bulk sampling

- Used in the absence of sand lenses or for indurated sediments
- **For indurated sediments-** an ~1 ft³ block of cohesive sediment should be wrapped with foil and duct taped to keep sample in tact
  - ◇ Middle of block used for D_E, outer used for D_R
- **For bulk sampling**- pit or exposure should be covered with light-proof tarp- then ~5 cm of surface (light-exposed) sediment excavated
  - ◇ Can be sampled on moonless night with red headlamps
- Transport samples in light-proof containers or bags

USU Luminescence Lab Photo
http://www.usu.edu/geo/luminlab/

Rocks- via slices (Dremel tool), small cores, night sampling

Rock art in Capitol Reef NP

Chaco Canyon National Historic

Ceramics

Minimum specimen dimensions: >5mm thick, >2cm in diameter

Ceramics and rocks heated to >450°C can be dated with TL or OSL

Ceramic sherd near Kanab, UT

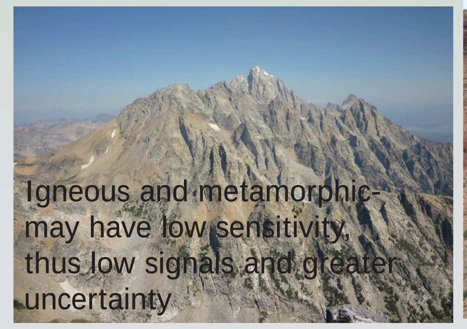
3) Considerations regarding D_E sample

Think **transport distance** and **depositional environment** as it relates to optical bleaching (exposure to sunlight)

What is the bedrock type from which the sediment is derived? This can affect the samples sensitivity to luminescence.

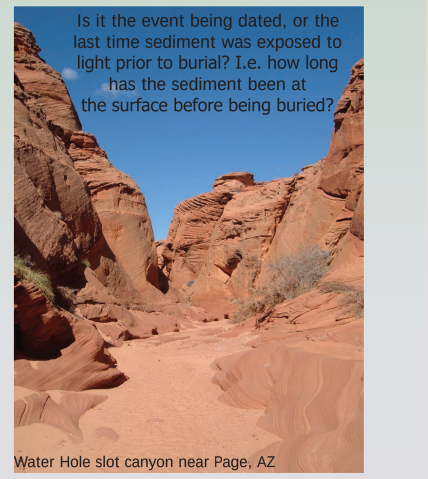

Bear Glacier near Hyder, AK.


Kanab Dunes, southern Utah



View of the Grand Teton from summit of Buck Mtn

Colorado River near Canyonlands NP



Water Hole slot canyon near Page, AZ

4) Collection and measurement of D_R sample

- Use trowel to sample sediment within **15-cm radius** of OSL tube, paying particular attention to sediment closest to tube location
- **Measure depth below geomorphic or land surface- noting recent natural or man-made erosion or surface accumulation**
- Gamma dose influence is spherical, collect sediment from back of OSL tube hole
- Do not use cloth bags as dry sediment can escape through material
- Make note of lithology, if coarse-grained representatively include larger clasts


OSL tube in alluvial sediments near Escalante, UT, circle outlines sample area for dose rate material.

Analytical methods - check with laboratory for preferred technique and availability

In the lab - determine concentration of radioelements:

- **ICP-MS analysis:** Fill 1-quart bag with sediment (100-200 grams needed after splitting in lab)- use Ziplock freezer or double bag
- Gamma decay counting - requires at 500 grams of sediment, good for coarse-grained deposits
- Alpha or beta decay counting - requires 70-100 grams of sediment
- Neutron activation analysis
- Atomic absorption spectroscopy

On site:

- Field-portable gamma spectrometer (Ge detector)
- Buried dosimeter


Joel Pederson (USU) collecting material for dose rate analysis

Average D_R values = 0.5 - 3.0 Gy/ka

5) Considerations regarding D_R sample

Homogeneous sedimentology and stratigraphy with minimal chemical and physical alteration is ideal


Soil pit in dunes near Kanab, UT

In reality, stratigraphy is often weathered and heterogeneous like in this paleoseismic trench


Trench exposure through faulted colluvium central VA

6) Misc. Notes on Artificial Dosing

- Typical baggage x-ray machine produces 2 µSv = 0.0000002 Gy dose. While we suggest ground transportation when shipping samples, air transportation likely has negligible effects on paleodose.
- NEVER bring D_E samples in carry-on or checked baggage.
- Sediment cores analyzed for porosity and density are usually exposed to gamma radiation from ^{137}Cs source. This has been shown to not affect the paleodose of the samples (Couapel and Bowles, 2006).

7) Test case: Grain size characterization and water retention modeling

1. Grain size analysis

- 2 g of dry, homogenized dose rate sediment suspended in de-ionized water
- Grain-size in % clay (0-3.9 µm), silt (4-62.9 µm) and sand (63-2000 µm) determined using a Malvern Mastersizer 2000 laser particle-size analyzer at Utah State University

Table 1. Grain size results and inputs to Rosetta Lite v.1.1 in Hydrus 1D

Sample ID	% clay	% silt	% sand
USU-115	3.49	21.36	75.15
USU-263	5.96	15.46	78.59
USU-361	3.16	9.36	87.48
USU-362	1.63	4.49	93.88
USU-365	1.49	3.18	95.33
USU-446	4.09	11.52	84.39
USU-519	8.01	32.31	59.68
USU-520	1.80	4.64	93.56

- Additional parameters to enter into Rosetta Lite v1.1 if available: bulk density, water content at 33kPa and water content at 1500 kPa

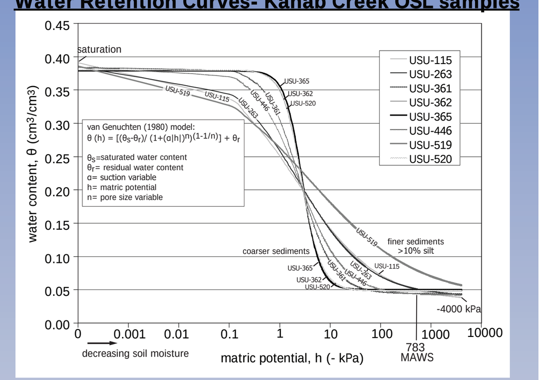
2. Rosetta Lite v 1.1 modeling to generate WRC

- Rosetta Lite v1.1 is a PTF, or predictive function used to translate basic soil data (texture, morphology, structure, etc.) into soil properties
- % size classes of sand, silt and clay were entered into the Rosetta Lite v.1.1 software program which is part of the freeware Hydrus 1D
- WRCs relate water content (θ) to soil water pressure, or matric potential (h), of the sediment (in -kPa or -cm H_2O)
- The WRC shape is influenced by capillary (primary force) and adsorptive forces within the soil matrix as they relate to the size, shape, and chemical composition of grain surfaces
- Generally, as matric potential becomes increasingly negative, soil water content decreases

Table 2. Outputs from Rosetta Lite v 1.1 in Hydrus 1D

Sample ID	θ_s (cm³/cm³)	θ_r (cm³/cm³)	α (1/cm)	n (-)	Ks (cm/day)
USU-115	0.393	0.0327	0.0446	1.5472	80.75
USU-263	0.386	0.0397	0.041	1.6261	84.77
USU-361	0.384	0.0439	0.0391	2.2971	237.76
USU-362	0.380	0.0487	0.0359	3.2514	639.5
USU-365	0.379	0.0501	0.0349	3.4957	784.83
USU-446	0.385	0.042	0.0404	1.9808	155.94
USU-519	0.390	0.0374	0.0242	1.4128	42.45
USU-520	0.380	0.0488	0.0358	3.1957	608.64

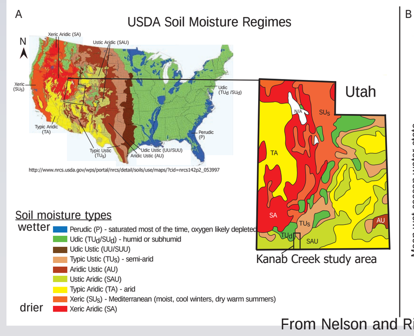
Water Retention Curves- Kanab Creek OSL samples

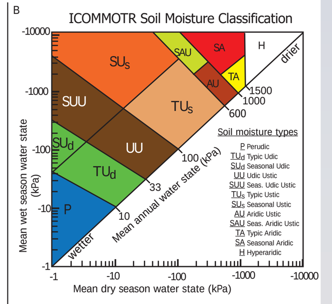


From Nelson and Rittenour (2015)

3. Determine mean annual water state (MAWS) matric potential

- Mean annual water state (MAWS) is the matric potential (soil moisture) of the mean wet season and mean dry season water states
- Find the soil moisture regime of geographic area on USDA-NRCS Soil Moisture Regime Map (A)
- Relate the soil moisture regime to ICOMMOTR classification (B) to determine the matric potential -read from the diagonal line
- Calculate the geometric mean from the upper and lower MAWS matric potentials read from the diagonal line





From Nelson and Rittenour (2015)

8) Water Content Measurements

In-situ

- Collect ~ 50 g of sediment in air-tight container, if needed ends of OSL tube or D_R sample can be used
- Measure immediately following sampling
- Gravimetric water content: obtain wet weight, dry in 100°C oven at least overnight, obtain dry weight
- If available, soil moisture probes can also be used
- All in-situ moisture content measurements can suffer from non-representative conditions at the time of sampling or effects of surface drying at the exposure front

Saturation

- If possible, collect samples in the field that preserve the original grain packing of the sediment

To retrospectively measure saturated water content from D_R sample:

- Homogenize and uniformly split into four 50 mL centrifuge tubes; each tube containing approximately 20g of sediment
- Weigh the dry sediment in each tube then slowly add de-ionized water to the sediment to fill pore spaces
- Centrifuge for 10 min at approximately 1600 rotations per min to allow natural settling and packing of the sediment for the purpose of approaching original field porosity
- Decant excess water and remove via suction, taking care not to lose any sediment
- Obtain wet weight for each tube and calculate gravimetric water content

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