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Thermoluminescence Dating Procedures In Use At The U.S. Geological Survey, Denver, Colorado

by

Hugh T. Millard, Jr.[1] and Paula B. Maat[1]

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[1] U.S. Geological Survey, MS 974, DFC, Box 25046, Denver, Colorado, 80225

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CONTENTS

APPENDICES

TABLES

FIGURES

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Hugh T. Millard, Jr. and Paula B. Maat

Abstract

The thermoluminescence (TL) dating method relies on traps in the crystal lattices of certain minerals (primarily feldspar and quartz) being cleared of electrons and holes by sunlight during transport and deposition of sedimentary materials. Following deposition and burial of these materials, the traps refill with electrons and holes through the action of natural ionizing radiation, i.e., the sample functions as a radiation dosimeter. The numbers of electrons and holes are a function of the radiation dose received by the sample and this is related to the length of time since burial. TL is one of several methods for determining the size of the population of electrons and holes. The age is computed using the very simple equation:

$$\text{Age} = \text{ED} / \text{DR},$$

where ED = radiation dose accumulated by the sample and DR = radiation dose rate seen by the sample during burial. In 1991, a decision was made to establish a thermoluminescence (TL) dating laboratory within the Branch of Isotope Geology to date Quaternary sediments for several programs in the U. S. Geological Survey. Since then, we constructed a dark laboratory, purchased and set up instruments, established a number of procedures, and collected over 100 samples at localities ranging from above the Arctic Circle to the southern United States.

Most of the procedures adopted for sample collection, sample treatment, separation of the fine silt fraction, plating of the fine silt on disks, normalization of the disks, and TL experiments and data reduction to establish ED are adapted from procedures described in the literature or existing in other laboratories. For example, we employ the widely-used total bleach and partial bleach methods on each sample to determine ED. We did apply three different methods to calibrate our beta irradiator because of its importance to the accuracy of TL dating.

For the determination of DR, we took advantage of the breadth of experience and techniques available within the Survey. We applied field gamma spectrometry, laboratory gamma spectrometry, and delayed neutron counting to each of the approximately 100 samples. This large sample population permits more precise and comprehensive intercomparisons among techniques than would be true for fewer samples. A comparison between delayed neutron counting and laboratory gamma spectrometry established that there is no systematic disequilibrium in the U and Th decay chains in these samples. The comparison between field gamma spectrometry and laboratory gamma spectrometry allowed the determination of moisture correction factors for field gamma spectrometry.

Finally, we applied the procedures to a set of 21 samples (12 loess, 6 sand, and 3 silt samples). Two of these samples gave ages which were at variance with their stratigraphic frameworks but the remainder displayed very good agreement. The appendices of this report list procedures in sufficient detail to function as a manual for our TL dating method.

Introduction

Radiation Exposure Geochronometers.

Thermoluminescence (TL) dating is one of several geochronometers which rely on exposure to low-level natural radiation to produce a measureable signal that grows with time. These techniques are named for the methods used to detect the signal. Two other techniques in this category are optically stimulated luminescence (OSL) dating and electron paramagnetic resonance (EPR) dating, also called electron spin resonance (ESR) dating. This brief introduction is not intended to substitute for the extensive reviews of these techniques which may be found in the literature. For example, Aitken (1985), Berger(1988), Forman (1989), and Wintle (1990) present very complete discussions of TL procedures, a recent discussion of OSL may be found in Aitken (1992), and the ESR technique has been described by Grun (1989a and 1989b) and Ikeya (1993).

These clocks require:

1) the presence of intrinsic crystal defects which are empty of electrons and holes at some zero time,
2) ionizing radiation to produce electrons and holes, a fraction of which migrate and become trapped at the defect sites,
3) sufficient stability for these trapped electrons and holes to remain in the traps at ambient temperatures over the geologic time range of interest,
4) a sensitive method (e.g., TL, OSL, or EPR) to measure the population of trapped electrons and holes,
5) a method of calibration that allows conversion of the measured numbers of trapped electrons and holes to equivalent radiation dose, ED, and
6) an accurate and reliable method for quantifying the strength of the natural radiation field, i.e., the radiation dose rate, DR.

These clocks have been applied primarily to the last few hundred thousand years, although attempts have been made to extend them back further. The practical result has been to extrapolate from the better established framework of radiocarbon chronology backwards in time.

When applied to dating sedimentary materials (usually sand and loess or other silt), these techniques rely on the following sequence of events:

1) During eolian or fluvial transport, mineral grains (primarily quartz and feldspar) are exposed to sunlight of sufficient duration and intensity to clear the defect sites of electrons and holes, i.e., the sample is bleached (the clock is zeroed).
2) Following deposition and burial, exposure to natural ionizing radiation gradually repopulates the defect sites with electrons and holes (the clock runs).
3) A signal, proportional to the concentration of trapped electrons and holes, is measured by TL, OSL, or EPR (the clock is read).
4) Increments of artificial radiation dose are added from a calibrated irradiator and the signal remeasured. Thus, the response of the sample is calibrated with respect to signal/unit of radiation dose and one can then convert the natural signal to equivalent radiation dose (ED).
5) Radiation dose is converted to time by dividing by the natural radiation dose rate seen by the mineral grains, i.e.,

$$\text{Age} = \text{ED} / \text{DR}$$

where ED = radiation dose seen by the sample = signal / (signal/ unit dose) and DR = natural radiation dose rate (e.g., dose/year or dose/kyr).

The natural radiation dose arises primarily from the decay of K-40, U-235 and it's daughters (U-235 decay chain), U-238 and it's daughters (U-238 decay chain), and Th-232 and it's daughters (the Th-232 decay chain). Cosmic rays and the decay of Rb-87 make lesser contributions. Thus, the radiation consists primarily of gammas (range = centimeters), betas (range = hundreds of micrometers), and alphas (range = tens of micrometers). One chooses the grain sizes and treatments for dating based on these ranges. For example, when 100-μm or 200-μm sand grains are used for these techniques, the surface exposed to external alphas is removed by etching and correction is made for the gradient in the external flux of betas. For TL dating, 4-11 μm silt is often used because all three types of radiation traverse grains of this size and the luminescence can easily escape to be detected.

Thermoluminescence Dating.

The 4-11 μm silt fraction separated for TL dating is mounted as a thin deposit on aluminum disks. We then carry these through the various experiments required to generate a value for ED. In addition to the stable signal, artificial irradiation may also produce a less stable TL signal which is responsible for the anomalous fading discovered by Wintle (1973 and 1977) and discussed by Aitken (1985, p. 274-281) and Berger (1988). The disks are preheated to remove this unstable signal. The TL instrument consists of an oven plate for heating the sample under conditions in which the temperature increases linearly with time, optical filters to attenuate photons from portions of the optical spectrum, and a photomultiplier to detect the remaining photons. Most of the signal is due to thermoluminescence from feldspar and quartz grains. The TL instrument records the signal as photons/5-degree C interval and plots the resulting glowcurve as TL signal vs. temperature. The glowcurve is actually a composite of Tl peaks from a number of different traps (see Aitken, 1985, p. 272 for a partial list of traps and their parameters).

A major complication in the TL dating technique arises from the fact that sunlight bleaching during transport of the sedimentary material does not zero the inherited TL signal completely. Several strategies have been adopted to compensate for this. We use the total bleach method (Singhvi et al., 1982; Aitken, 1985, p. 222) and the partial bleach method (Wintle and Huntley, 1980; Aitken, 1985, p. 225). We do not use the regeneration method (Wintle and Huntley, 1980; Aitken, 1985, p. 223) because it appears to underestimate the ages of older loess (e.g., see Rendell and Townsend, 1988). In addition, we perform experiments on selected samples to:

1) establish the degree of sunlight bleaching as a function of time in order to validate the sunlight bleaching times chosen for the total bleach and partial bleach methods, and
2) to determine whether or not the preheat treatment removes the less stable signal generated by artificial irradiation.

Application to Loess, Sand, and Material Related to Earthquake Events.

Pye (1987, p.199) defines loess as "... a terrestrial windblown silt deposit consisting chiefly of quartz, feldspar, mica, clay minerals and carbonate grains in varying proportions." Ranges of values for the physical properties of loess are given in Table 1. Unfiltered sunlight bleaches the mineral grains during transport, thereby resetting the TL clock. Because glacial processes are thought to be the chief suppliers of the silt which results in loess, ages determined for loess layers are used to define the timing and duration of events in the paleoclimate record.

We have sampled loess from vertical sections in quarries, landfills, road cuts, railroad cuts, stream cutbanks, or soil pits dug in thin loess caps. The deep vertical fissures, which "commonly develop sub-parallel to the face of high, steeply-inclined loess sections as a result of tensile stresses in the upper part of the slope" (Pye, 1987, p. 222), provide pathways for the introduction of younger loess from above. The vertical face must be removed sufficiently to get behind these cracks and their contamination.

Table 1. Physical properties of loess (Pye, 1987, pp. 207 and 219).

porosity	40 - 55 %
moisture content	10 - 25 %
bulk density, dry	1.1 - 1.5 g cm^{-3}
dry, weathered	1.5 - 2 g cm^{-3}
bulk density, typical	1.5 - 1.8 g cm^{-3}
specific gravity of mineral grains	2.1 - 2.75
predominant grain size, unweathered	10 - 50 μm

We have sampled sand by augering down into the tops of dunes or sand sheets, or from vertical sections dissected by quarries, road cuts, or stream cutbanks. Most of the silt in sand appears to be depositional (e.g., see Muhs, 1985).

Trenches dug across earthquake faults provide opportunities to sample wash facies from distal portions of fault-related colluvial wedges, eolian silt layers, and A horizons (Forman et al., 1991). Ages for these samples are used to estimate recurrence intervals for earthquakes on the faults.

Sample Collection

The primary objective is to obtain a stratigraphically significant sample from which sufficient 4-11 μm silt can be separated to conduct the TL experiments. We avoid extensive bioturbation, contamination with silt material introduced from above or below, intense pedogenesis, and evidence of oxidizing or reducing conditions which would lead to migration of radioelements or their daughters in the decay chains. Finally, we prefer situations in which it is possible to make reasonably accurate estimates of the moisture content over the period of burial.

Appendix A contains detailed sample collection procedures. Sampling positions are chosen on vertical exposures which are as free as possible of cracks, roots, krotovina, carbonate, and oxidation streaks. The sample for TL dating is collected from the rear of a 1-foot deep augered hole by pounding in a sample tube with a "drive tube" and sledge hammer. The sample tube is capped as it is removed so that no light reaches the sample and stored in a black, opaque film bag until processed. Later the 4-11 μm silt fraction is separated from this sample for the TL experiments which estimate the equivalent radiation dose (ED) to the sample.

The sample hole is then augered to a depth of 2 feet, a bulk sample taken from the augered material and double bagged to prevent loss of moisture, but not protected from exposure to light. Later, this sample is used to measure moisture content at the time of collection and may also be used to establish saturation moisture content, and the concentrations of K, U, and Th by laboratory gamma spectrometry and delayed neutron counting.

In situ gamma spectrometry is performed using a portable gamma spectrometer with a 3 x 3-inch sodium iodide detector inserted 20 inches (50 cm) into the sample hole. This location places the gamma detector close to the positions of the two samples. Measurements on the bulk sample and field gamma spectrometry are used to compute the radiation dose rate (DR). See Appendix B for procedures describing operation of the portable gamma spectrometer and the section "Determination of Concentrations of K, U, and Th" for discussion of the data and methods of computation.

Where buried soils are encountered, the following simple model is assumed:

A horizon - This soil horizon forms first but is often eroded away. If present and formed in an arid environment, the top of this horizon is acceptable for TL dating unless contaminated by younger clay and silt filtering down from above. The presence of clay films is evidence of such contamination. The mineral grains are repeatedly exposed to sunlight through bioturbation and therefore, the TL age corresponds to the time of formation of the soil.

B horizon - This horizon forms after the A horizon and is less suitable for TL dating. If it is high in clay, it can swell and shrink and thus, is often comtaminated with younger silt filtering down from above via roots and cracks. The radiation history is less certain because water moving through the horizon may creat oxidizing or reducing conditions.

C horizon - This horizon usually provides the most reliable TL ages. Those from the bottom of the horizon date the start of deposition.

For a surface locality, vertical augering is performed. The sample for TL dating is collected from the C horizon by filling the auger bucket, withdrawing it to the surface, and pushing the sample tube into the material from the rear of the bucket. Care is taken throughout to protect the material in the bucket from exposure to light. The bulk sample is also collected from material in the bucket. In situ gamma counting is performed by lowering the detector into the augered hole on a steel cable.

Treatment of Bulk Sample

Figure 1 shows a flow diagram for treatment of the bulk sample. See Appendix C for details of the procedures. The moisture content of each sample is determined by weighing the bulk sample, allowing it to dry in air for several days, and then reweighing. Of course, this value for the moisture content applies only to conditions at the time of sample collection, when field gamma spectrometry is performed, and may have varied considerably over the period of burial. Thus, we arbitrarily assign coefficients of variation of 10% to the moisture content used to compute the correction to field gamma spectrometry and 30% to the moisture content used to compute dose rate over the period of burial.

Any pebbles or twigs are removed as the sample dries and their weight subtracted from that of the sample. In order to promote sample homogeneity, clumps are broken up by hand during drying. If hard clumps have formed, the sample is passed through a jaw crusher with ceramic plates reserved for soft sedimentary materials. If necessary, we repeat this grinding until the sample passes through a No. 10 sieve (<2-mm size particles).

The saturation moisture content may be established by mixing excess water with a weighed portion of the dried sample (~15 grams), allowing it to settle, centrifuging, and removing the supernatant. Reweighing establishes how much water has been retained. The compaction achieved by centrifugation is a function of time and the rate of rotation. 4000 rpm in our clinical centrifuge is equivalent to a force of about 1600xG or about 13 m of overburden (assuming a dry density of 1.5 g/cm3). This force is sufficient to collapse the pore space in the loess if the loess is saturated with water (Rendell, 1985b and Pye, 1987, p. 219) and results in a reduction in the saturation moisture content of about 10% to 15%. Five minutes of centrifugation is long enough to approach the asymptotic limit of compaction for both loess and sand (see figure 2). It would be more realistic to choose settings for the centrifuge which correspond to the overburden observed in the field (see Table 2). The saturation moisture content represents an upper limit to the true moisture content over the period of burial. The moisture content of sediments above the water table will fluctuate over a range of values less than the saturation moisture content.

Laboratory gamma spectrometry may be performed on 600 grams of the bulk sample. We seal this counting sample for at least two weeks prior to counting to allow buildup of radon. U and Th may also be determined by delayed neutron counting on a 10-gram aliquot of the bulk sample. Discussions of these techniques and the data they generate will be deferred until the section "Determination of Concentrations of K, U, and Th". Details of the procedures may be found in Appendices C.4 (laboratory gamma spectrometry) and C.5 (delayed neutron counting).

Record Keeping and Flow of Data

Because numerous operations must be performed on each sample and several samples are carried through the procedures at a time, relatively detailed records must be kept. We organized the record keeping and the flow of data so as to minimize the burden on the experimenter. Appendix D contains an outline of the system and examples of the individual forms.

Calibration of Centrifuge

We calibrated the clinical centrifuge (see figure 3) using a tachometer in the laboratory of Rolf Kihl at the University of Colorado. The results are shown in Table 2. We use the following equation to compute RCF (relative centrifugal force) (Jackson, 1973. p. 127):

$$\mathrm{RCF(xG)} = (1.118\mathrm{e}{-5}\ \mathrm{min}^2/\mathrm{cm}) \times (\mathrm{rpm})^2 \times R$$

where R = radius of rotation (cm). The equation to compute the approximate overburden depth of 1.5 gm/cm3 loess to produce collapse of the underlying loess is:

$$\mathrm{depth\ (cm)} = (6.7\mathrm{e}{-5}\ \mathrm{min}^2) \times (\mathrm{rpm})^2$$

Table 2. Calibration of clinical centrifuge (IEC, Model CL, S/N 89752M-3).

			RCF(xG)					
Setting	RPM	Time to Come Up to Speed	R= 6 cm	7 cm	8 cm	9 cm	10 cm	Corresponding Overburden Depth, meters
1								
2	50							
3	1390		130	151	173	194	216	1.3
4	1750		205	240	274	308	342	2.1
5	2550		436	509	582	654	727	4.4
6	3050		624	728	832	936	1040	6.2
7	4000	55 sec	1073	1252	1431	1610	1789	10.7

Separation of Silt from Sample in Sample Tube

The procedures used to separate the 4-11 μm fine silt from the sample for TL dating are taken from training Steven Forman gave to one of us (PBM), Aitken (1985), and Jackson (1973). Figure 4 shows the flow diagram for treatment of the sample in the sample tube. See Appendix E for details. Briefly, a 100-gram sample from the center of each sample tube is processed to separate the 4-11 µm fine silt fraction for TL dating. Each sample is treated with 4 N HCl to remove carbonate, 30% hydrogen peroxide to destroy organic material, and dispersed in Na pyrophosphate solution. The 4-11 µm size fraction is separated by sieving and Stokes settling in water under gravity and centrifugation (see Table 3). It is then plated on 1-cm diameter aluminum disks from a suspension in methanol. These procedures must be performed under reduced lighting conditions to minimize artificial bleaching of the samples. We adjust the intensity of our sodium vapor lights, which emit a single wavelength (589 nm), to provide the requisite sensitivity for the human eye but still not bleach the samples.

Table 3. Particle settling in water under the force of gravity or centrifuge at 20° and 26°C. The rate of settling is given for gravity and the time to settle through 4.5 cm for the centrifuge. Viscosity of water = 0.01005 poise at 20°C and 0.00874 poise at 26°C. Specific gravities = 2.65 for the particles and 1.00 for water.

Particle Diameter	Gravity Settling	Centrifuge (1390 RPM)
63 µm	2.5-2.8 sec/cm	
11 µm	80 - 93 sec/cm	2.5 - 2.2 sec
4 µm	10 - 12 min/cm	19. -17. sec

Table 4. Numbers of disks required for TL experiments. Abbreviations represent:

N = natural sample disk
SL = sunlight
PH = preheat
B = beta irradiation
1 = most samples from a region
2 = one sample from each region

Experiment			Sample Category 1	2	Data Points
Sunlight bleach sensitivity	(SL)	=		36	(N), 8*(N, h SL)
Test for anomalous fading	(AF)	=		16	(PH,Stored), (no PH,Stored), (PH,Immed), (no PH,Immed)
Total bleach TL method	(TB)	=	24	24	(N,16h SL), (N), 4*(N+ mB)
Partial bleach TL method	(PB)	=	20	20	(N,1h SL), 4*(N+ mB,1h SL)
Total disks			44	96	
Prepare			72	96	

Preparing and Normalizing Disks for TL

See Appendix F for detailed procedures. The capacities of the containers used to hold the disks during preparation and storage of disks:

vial block = 18 x 4 = 72 shell vials
cell well box = 6 x 4 = 24 disks (four boxes per sample)

Because of imprecision in the various parameters involved in TL measurements, the readings from four disks must be averaged for each data point in the TL experiments. Thus, four times as many disks must be prepared as there are data points. The numbers of disks required for the various TL experiments are shown in Table 4.

Normalization of Disks by IRSL.

The disks are normalized for intercomparison (i.e., to compensate for disk-to-disk variation) by a 5-second exposure to infra-red illumination (Aitken and Xie, 1992) and detection of the resulting luminescence by a photomultiplier covered with Schott BG-39 and Kopp 7-59 filters (as well as a Pyrex window). This procedure, which is called infra-red stimulated luminescence (IRSL), is a special application of OSL. The TL signal is reduced by about 2% by this treatment. The reproducibility of the amount of sample on each disk, as reflected in the normalization factor, varies widely among samples (see Table 5).

Table 5. Distribution of normalization factors for typical samples.

Sample	Number of Disks	Range of Normalization Factors	Percentage of Disks Within Ranges 0.9-1.1	0.8-1.2
Loess samples:				
MMV-14	51	0.319 - 1.450	18 %	42 %
MMV-26	38	0.678 - 1.122	91	98
CPL-6	55	0.565 - 1.666	27	53
CPL-9	55	0.701 - 1.320	42	82
Silt samples:				
YVC-1	96	0.758 - 1.167	71	97
YVC-2	80	0.809 - 1.275	70	95

Normalization of Disks by Reirradiation and Glowing.

After performing the TL experiments and primary glows, those sets of disks not normalized by IRSL can be normalized by reirradiating for 5 minutes in the beta irradiator and reglowing using Schott UG-11 and Corning CS1-75 filters or other appropriate filters.

Calibration of Beta Irradiator

See Appendix G for the procedures to operate the beta irradiator. Calibration of the beta irradiator is necessary so that the artificial radiation doses delivered to the sample disks are known precisely. The accuracy of the equivalent dose, and thus that of the age itself, depends on this calibration. The Daybreak Model 801 multiple sample beta irradiator contains an Amersham Type

SIF1177 100-mCi strontium-90 beta source. The dose rate delivered by the irradiator was measured by three different techniques:

1) comparison to a calibrated gamma irradiator using CaSO4:Dy disks (Teledyne, Inc.),
2) reading of GafChromic foils irradiated in our beta irradiator (Christopher Soares, NIST), and
3) comparison to a calibrated beta irradiator at Ohio State University using CaF2 disks (Steven Forman, Ohio State University).

Table 6 shows the results of these calibrations and the weighted mean dose rate, 2.77 ± 0.04 Grays/minute. This value applies only to samples on aluminum disks. The dose rate for stainless steel backing is 3.71 Grays/minute.

Table 6. Summary of dose rates to samples on aluminum disks found during calibration of the Daybreak beta irradiator. All dose rates corrected for decay to January 1, 1993.

Method	Grays/minute	(CV)
Teledyne CaSO4:Dy disks	2.79 ± 0.15	(5.2%)
NIST GafChromic films	2.88 ± 0.14	(5.0%)
OSU CaF2 disks	2.76 ± 0.05	(1.8%)
Weighted Mean (on 1/1/93) =	2.77 ± 0.04	(1.6%)

TL Instrument

See Appendix I for detailed procedures for operating the TL instrument and glowing a set of sample disks. Figures 5 and 6 show schematic diagrams of the Daybreak Model 1100 automated TL instrument. A vacuum pump, nitrogen cylinder (with pressure regulator and flow meter), and a PC are external to the instrument and are not shown. The top section (photomultiplier/filter/IRSL assembly) can be lifted off the base (which contains the sample carousel, TL oven, and electronics) to load and unload sample disks. The following operations of the instrument are controlled by the TLAPPLIC software running on the PC and are common to both the TL and IRSL modes of operation:

1) sample changing,
2) switching the photomultiplier high voltage on and off, and
3) collecting counts from the photomultiplier and storing them in a multi-channel scaler.

Additional operations for the TL mode include:

4) switching vacuum on and off,
5) switching nitrogen purge gas on and off, and
6) raising the temperature of the TL oven along a ramp.

The one additional operation for the IRSL mode involves switching the infra-red light emmitting diodes (LED's) on and off. The operating parameters for the TL mode of operation are:

1) a ramp rate of 5°C (or 10°C) per second,
2) nitrogen environment,
3) maximum temperature of 500°C,

4) Schott UG-11 and Corning CS1-75 filters (samples MMV-5,6,7,9) or Schott BG-39 and Kopp 7-59 filters (samples NEC-17,18,19,20; NVP-2; MCS-3a,3b; MVL-2,3,4), and
5) background glowcurve taken after each primary glowcurve.

The operating parameters for the IRSL mode of operation (used to normalize disks) are:

1) 20 ma current to the infra-red LED's,
2) 1-second dwell time per channel for multi-channel scaler,
3) 5-second total time for multi-channel scaler,
4) sample temperature of 30°C, and
5) one background count taken before each set of samples is run.

TL Experiments

See Appendix H for an outline of the TL experiments. Four experiments may be performed on each sample. Two of these, involving determination of the rate of bleaching by natural sunlight (Aitken, 1985, p. 221-222 and p. 231-233) and susceptibility to anomalous fading (Aitken, 1985, p. 274-281), are done on at least one sample from each locality. The other two experiments, involving dating by the total bleach method (Singhvi et al., 1982; Aitken, 1985, p. 222) and the partial bleach method (Wintle and Huntley, 1980; Aitken, 1985, p. 225) are performed on all samples. In the future, we recommend performing the susceptibility to anomalous fading experiment on every sample.

The determination of sunlight bleach sensitivity establishes the efficiency of sunlight for bleaching a sample. The fraction of TL signal remaining at various TL temperatures is plotted against the sunlight bleach time (see figure 7). In the simplest interpretation of these graphs, a rapidly-bleached component yields the initial rapid decrease in TL signal. Later, a slowly-bleached component dominates the decay. The family of curves is used to determine the best sunlight bleach times for the total bleach and the partial bleach methods.

The test for anomalous fading establishes the effectiveness of the preheat treatment for removing the less stable TL component generated by beta irradiation. It also can be used to eliminate those samples which are extremely unstable and therefore unsuitable for TL dating. Where used, the preheating conditions are 124°C for 64 hours followed by several weeks of storage.

In the total bleach TL method (see figure 8), a growth curve is generated by irradiating natural disks for various lengths of time. We choose the longest of these times to produce about 8x our best estimate of the ED for a particular sample. The resulting TL signals are plotted against the radiation dose. This curve is then extrapolated to it's intersection with a residual level baseline defined by the TL signal from a natural disk exposed to sunlight for 16 hours. The position of this intersection on the dose axis defines the equivalent dose (ED). Unfortunately, the shape of the growth curve is not known precisely over this interval and much effort has been devoted to overcoming this problem (for recent examples, see Prescott et al, 1993 and Sanzelle et al, 1993). Most solutions are variations of the following three approaches:

1) the curve is assumed to be a straight line (for example, young samples which received small radiation doses),
2) the curve is assumed to be a saturating exponential (older samples which received large radiation doses), or
3) the shape of the curve is "reconstructed" by the regeneration method.

The true picture is more complicated and each assumption has its limitations. We use approaches 1 and 2.

Figure 9 illustrates the partial bleach method. The growth curve from the total bleach method is combined with a second growth curve generated by irradiating another set of natural disks for the same periods of time but also exposing them to sunlight for a period of time chosen to remove the rapidly-bleached component. The intersection of these two growth curves again defines an equivalent dose on the dose axis. As with the total bleach method, uncertainty in the shapes of the growth curves introduces error into the determination of equivalent dose. However, the error is greater than the total bleach method because two curves must be extrapolated rather than one.

TL Data Reduction and Computation of Equivalent Dose

See Appendix I.3 for details. We reduce the glowcurve data using the Daybreak 1100SI TL Applications software (Bortelot, 1990). Briefly, background curves are subtracted from each glowcurve, curves are shifted to compensate for temperature mismatches among disks, and normalization factors for each disk are entered (figures 10 and 11). Then, exponential growth curves are generated (i.e., TL signal vs. radiation dose, see figure 12) and equivalent dose (ED) vs. temperature curves plotted
(see figure 13).

Sometimes, we must merge or reorganize files before reducing the data. For example, the total bleach data must be merged with the partial bleach data to complete the partial bleach data file. Temperature shifting of glowcurves must be performed with care. Ordinarily, the glowcurve maxima for the different groups of four disks do not lie directly under one another. They usually lie along a smooth curve arching from lower temperature for disks with the highest radiation doses to a higher temperature for the natural disks and a still higher temperature for the sunlight bleached disks (see figures 10 and 11). This progression is a reflection of the changes in the relative contributions of different peaks to the glowcurves. Construction of this curve can provide guidance for shifting maxima within groups where they are badly scattered.

The software computes a growth curve for the data points generated by passing a vertical line (single temperature) through the family of glowcurves. A least squares fit is made either to a line passing through the points or, for the saturating exponential, to a line passing through a logarithmic transformation of the data. An iterative algorithm is employed for the latter fit. The natural (unirradiated) disks are given double weight. The Daybreak software estimates errors for the ED's using Rendell's equation 1 for the regression of TL signal on dose (Rendell, 1985a). The saturating exponential growth curve shown in figure 12 for the total bleach method at 380°C is relatively well behaved in that the extrapolation to find equivalent dose is short and the uncertainty in equivalent dose is small. However, for the partial bleach method, we must find the intersection of two saturating exponential growth curves and the equivalent dose usually is much less certain. When the two curves meet at a small angle (the partial bleach is not long enough) this uncertaintly may become even larger.

The equivalent dose vs. temperature curve in figure 13 is constructed from growth curves for all temperatures. The objective is to find a temperature region over which the change in the equivalent dose is minimal, i.e., a plateau in the curve. We conclude that within such a temperature domain, the glowcurves are exhibiting acceptable TL dating behavior. The traps corresponding to that temperature regime produce a TL signal which truly reflects the radiation dose they have received. This is true whether the dose was received over geologic time (the natural disks) or over short periods of time (the artificially irradiated disks). The sample is behaving as a good dosimeter. Unfortunately, the example shown in the figure is not typical. Often the plateau is narrower. Sometimes only an inflection or a maximum in the curve is obtained. Experience and judgement can play a large role in choosing the equivalent dose for a sample. We use agreement between the total and partial bleach ages as a criterion for acceptable data (for example, see Berger, 1988).

Determination of Concentrations of K, U, and Th

We determine the concentrations of K, U, and Th, from which natural dose rates are computed, from in situ gamma spectrometry performed with the Exploranium GR-256 portable gamma spectrometer and a 3 x 3-inch sodium iodide detector. This instrument is calibrated using the Department of Energy calibration pads in Grand Junction, Colorado.

The effects of radon migration, attenuation of radiation by moisture, and disequilibrium within the radioactive decay chains may show up in three different places in our TL procedures:

1) calibration of the portable gamma spectrometer - We assume the moisture content of the pads is the same as during their calibration by DOE (George et al., 1985). Because of the long counting times required, radon "exhalation" was not determined by DOE on these pads and we are forced to ignore it. Otherwise, equilibrium within the U-238 decay chain above radon should be excellent.

2) gamma spectrometry in the field - We determine the moisture content of the bulk sample. Radon migration and disequilibrium are not directly addressed. However, we select sites where chemical migration of daughter products is less likely and we take gamma spectra immediately after augering the sample hole in order to reduce the effects of radon migration on in situ gamma spectrometry.

3) during calculation of TL dose rate - A reasonable estimate is made of the moisture content through geologic time with the understanding that this estimate carries a large uncertainty. Unless conditions at the time of sample collection are unusual (e.g., sustained rain over a long period of time) the moisture content in the collected sample is used. We ignore radon migration and disequilibrium but we avoid cracks and chemically altered sites as much as possible.

The effects of these factors were investigated and, where possible, we attempted to compensate for them. First, we compared the concentrations of U and Th found by laboratory gamma spectrometry on 600-gram splits of the bulk sample to those found in 10-gram aliquots of the bulk sample by the very different analytical technique of delayed neutron counting. The effect of radioactive disequilibrium on gamma spectrometry would contribute to a lack of agreement. Then the concentrations of K, U, and Th found from field gamma spectrometry (uncorrected for moisture content) were compared to the laboratory gamma spectrometry values. Here, moisture and radon migration would contribute to a lack of agreement. We arbitrarily attributed discrepancies to the missing moisture correction and computed a value for this correction as a function of moisture content. Finally, the moisture corrected concentrations from the field gamma spectrometry were recompared to the concentrations from the laboratory gamma spectrometry and the degree of improvement noted.

Delayed Neutron Counting for U and Th.

See Appendix C.5 for detailed procedures. This technique relies on the fact that U and Th in a sample undergo induced fission in a nuclear reactor and their fission products continue to emit delayed neutrons after the sample is withdrawn from the reactor (Amiel, 1962). The sample is irradiated in the Geological Survey TRIGA Reactor (GSTR) for 1 minute in a cadmium-lined terminus, removed from the reactor, allowed to decay for 1 minute, and the delayed neutrons counted with an array of BF_3 detectors. This process is repeated with a bare terminus in the reactor. We use the delayed neutron counts produced by the two very different irradiations to compute the concentrations of U and Th in the samples. We include suitable standards. The technique lends itself to automation. In the U.S.G.S. delayed neutron system (Millard and Keaton, 1982; McKown and Millard, 1987), samples are loaded into magazines, these placed on sample changers attached to the GSTR pneumatic transfer tubes, and the entire system controlled and data collected with a PC program. We reduce the data using a VAX computer.

Moisture content and radon migration are not factors in the delayed neutron technique because the bulk sample has been dried and the concentration of U is determined directly. The precision for Th is poorer than that for U (see figure 14). We monitor the entire system by running aliquots of the Pikes Peak granite (5.88 ppm U and 30.2 ppm Th) with the samples. The precision for determining U and Th in replicate runs of this granite are 5% and 7% respectively. The small excess uncertainties over those in figure 14 are probably due to a lack of homogeneity within the granite powder.

Laboratory Gamma Spectrometry for K, U, and Th.

See Appendix C.4 for details of the procedure. Moisture and radon migration are not factors because the bulk sample has been dried and the sample is sealed and radon allowed to equilibrate before counting. We collect gamma spectra and then fit standard spectra of K, U, and Th to them using the least squares criterion. A computerized version of the graphical method described by Bunker and Bush (1966 and 1967) is used. We determine the three elements in standard materials to provide quality assurance. Comparisons between the laboratory gamma spectrometry values and the literature values for these standards (figures 15, 16, and 17) show excellent agreement. Most of the scatter displayed can be attributed to counting statistics. From these graphs, the coefficients of variation (CV's) for concentrations determined by laboratory gamma spectrometry are estimated to be 3% for K, 6% for U, and 3% for Th.

Field Gamma Spectrometry for K, U, and Th.

See Appendix B for procedures used to operate the portable gamma spectrometer. A number of good descriptions of in situ gamma counting can be found in the literature (e.g., see Wollenberg and Smith, 1964 or Duval, 1989). As with laboratory gamma spectrometry, the spectra could be fitted with standard spectra if care were taken in collecting the latter. Instead, most field gamma spectrometry employs the window approach and we did as well. The entire spectrum is collected but only the counts in windows surrounding the full energy peaks of K (1.46 MeV from K-40), a daughter of U-238 (1.76 MeV from Bi-214), and a daughter of Th-232 (2.62 MeV from Tl-208) are used in computations. Moisture content and radon migration must be addressed.

The counter backgrounds in each window are ordinarily evaluated from counting performed over water assumed to contain negligible concentrations of the three radioelements. The water should be at least 2 meters deep and the shore about 1000 meters distant in order to eliminate any contribution from nearby rocks and sediments. If the rocks and sediments in the vicinity are major sources of radon, Bi-214 in the atmosphere will contribute to the background. Under proper circumstances, only the counter itself (primarily the NaI detector), the boat, and cosmic rays contribute to the background. We measured the background at sea level on Wassaw Sound near Savannah, Georgia, thus minimizing the cosmic ray contribution. Table 7 shows a comparison between our results and the backgrounds supplied by the manufacturer with our spectrometer. The Savannah counter backgrounds are consistently lower than the manufacturer's.

Table 7. Comparison of backgrounds taken near Savannah, Georgia to the manufacturer's backgrounds.

	Background, cps	
Window	at Savannah, Georgia	Exploranium, Inc
K	0.540 ± 0.040	0.707
U	0.185 ± 0.011	0.207
Th	0.116 ± 0.008	0.180

The spectrometer was calibrated for 2-pi geometry by counting on the five Department of Energy calibration pads at Walker Field in Grand Junction, Colorado. Steele and George (1986) and George et al. (1985) give the concentrations and moisture contents for each of these pads. Because these pads are not protected from the weather, radon migration, precipitation enhancement, and fluctuations in the moisture content may be problems in their use. In Appendix J we show the derivation of the equations used for the spectrum stripping and simultaneous equation methods for treating calibration data and the correspondence between coefficients determined by these two techniques. We use the simultaneous equation method. The background concentrations for K, U, and Th found by this technique are quite large, presumeably because these backgrounds include the residuals and some of the pads contain high concentrations of the radioelements. Therefore, we follow a procedure in which the counter background for each window is subtracted from the counts in that window before solving the simultaneous equations for the coefficients. We then ignore the concentration backgrounds when using the equations to compute concentrations of the radioelelements from counting done in the field. The results for two different calibrations performed on these pads are shown in Table 8 along with the manufacturer's original calibration coefficients. The agreement among these calibrations is not very good. The primary coefficients for K and U for the 12/91 and 11/93 calibrations agree to within the quoted CV but the difference for Th is twice the CV. The precision and confidence in these calibration coefficients can only be improved by using more suitable calibrated pads or by conducting the calibration several times a year on the DOE Grand Junction pads and thereby accumulating a body of data and experience.

These coefficients are for a 2-pi counting geometry. They must be divided by 2 to obtain the coefficients for the 4-pi geometry used in our field gamma

Table 8. Manufacturer's 2-pi calibration coefficients and two sets of 2-pi calibration coefficients generated on the DOE's calibration pads in Grand Junction, Colorado. cK, cU, and cTh are cps in each window after background subtraction. Calibration constant units = concentration (percent or ppm)/cps.

Exploranium, Inc.:

$K = (0.297)*cK - (0.243)*cU - (0.041)*cTh$

$eU = (0.0090)*cK + (2.51)*cU - (1.69)*cTh$

$eTh = (0.065)*cK - (0.239)*cU + (8.13)*cTh$

Grand Junction, 12/91:

$K = (0.258 \pm 2.6\%)*cK - (0.242 \pm 4.2\%)*cU - (0.026 \pm 47\%)*cTh$

$eU = (3.23 \pm 5.9\%)*cU - (2.25 \pm 7.7\%)*cTh$

$eTh = (6.97 \pm 3.1\%)*cTh$

Grand Junction, 11/93:

$K = (0.266 \pm 2.6\%)*cK - (0.260 \pm 4.0\%)*cU - (0.009 \pm 141\%)*cTh$

$eU = (3.48 \pm 6.0\%)*cU - (2.56 \pm 7.8\%)*cTh$

$eTh = (5.98 \pm 3.3\%)*cTh$

spectrometry. A rough check of these 4-pi coefficients was performed by counting in one of the DOE vertical holes. Unfortunately, high concentrations of the radioelements in the enriched zone of this hole produce high counter dead times but the results in Table 9 seem to confirm our calibrations for U and Th. The agreement for K is poor and we have no explanation for this discrepancy. Again, the experiment needs to be repeated periodically.

Table 9. Comparison of concentrations from 4-pi counting on 12/91 in the DOE KW Model Subsurface Calibration Facility in Grand Junction, Colorado to literature values (computed from Steele and George, 1986, p. A-12).

Element	U.S.G.S.	Literature
K	11.2 %	4.6 ± 0.1 %
eU	306. ppm	361. ± 6 ppm
eTh	254. ppm	245. ± 4 ppm

Table 10. Results of counting with the portable gamma spectrometer in the "standard" location in the hallway of Building 15 at the Denver Federal Center. All counts were collected for 999 seconds and 5 or 6 countings were performed on each date.

Date	Battery Voltage	Counts in Window (mean of 5 or 6 countings, CV is for single count) K	U	Th
9/13/92	7.9	15,100 ± 1.2%	3625 ± 1.8%	3180 ± 2.7%
9/18/92	8.3	15,243 ± 0.9%	3741 ± 2.4%	3159 ± 1.5%
10/18/92	8.2	15,167 ± 1.0%	3728 ± 1.5%	3153 ± 1.0%
10/26/92	7.6	15,320 ± 2.4%	3732 ± 1.0%	3162 ± 1.8%
11/18/92	7.3	15,290 ± 0.8%	3898 ± 3.1%	3385 ± 2.0%
6/15/93	8.3	15,153 ± 1.1%	3742 ± 1.8%	3166 ± 1.0%
7/ 4/93	7.7	15,139 ± 0.9%	3685 ± 0.9%	3151 ± 1.7%
11/29/93	8.5	15,385 ± 0.5%	3721 ± 1.4%	3277 ± 2.1%
12/ 1/93	8.2	15,173 ± 0.9%	3699 ± 1.2%	3129 ± 1.2%
Mean		15,219	3730	3196
Range of CV's for single count		0.5-2.4%	0.9-3.1%	1.0-2.1%
Average CV for single count		1.1%	1.7%	1.7%
CV for each set of 5 or 6 countings		0.6%	2.0%	1.0%

Each in situ gamma counting in the field is performed twice. These duplicate determinations can be used to estimate counting precision as a function of concentration. The results are shown in figure 18.

A "standard" location in the hallway of Building 15 at the Denver Federal Center serves to provide quality assurance. "Concentrations" for this 3-pi locality are 2.0 % K, 3.2 ppm eU, and 15 ppm eTh. Counting is performed before and after each field trip to make sure the counter characteristics have not changed. The results over a period of 14 months are shown in Table 10.

Intercomparisons of Techniques and Evaluation of the Moisture Correction Factors.

The following intercomparisons are based on 80 to 100 samples. Most of these samples are loess. The agreement between delayed neutron and laboratory gamma counting values for U and Th is shown in figures 19 and 20. Although there is considerable scatter, the fitted lines lie sufficiently close to the 45-degree (perfect agreement) lines that we are unable to detect any significant bias (greater than about 10%) between these two techniques. Thus, although a few individual samples may not be in equilibrium, there is no systematic disequilibrium evident in the U and Th decay chains.

There is significant bias between laboratory gamma spectrometry and field gamma spectrometry when the latter are not corrected for moisture (figures 21, 22, and 23). The disagreement between the fitted and 45-degree lines for K and Th is about 30%. The equation describing the attenuation of gammas by water in a sample may be developed as follows (adapted from George et al., 1985, p. 42):

$$\frac{[\mathrm{CRdry}]}{[\mathrm{CRwet}]} = \frac{[\mathrm{LACOEFwet}]}{[\mathrm{LACOEFdry}]}$$

where CR = count rate, LACOEF = linear attenuation coefficient (1/cm) for a gamma of a particular energy, and dry and wet refer to the dry sample and wet sample respectively. Because MACOEFF = LACOEFF/D, where MACOEFF = mass absorption coefficient (cm2/gm) and D = density (gm/cm3),

$$\frac{[\mathrm{CRdry}]}{[\mathrm{CRwet}]} = \frac{[\mathrm{MACOEFwet}]\,[\mathrm{Dwet}]}{[\mathrm{MACOEFdry}]\,[\mathrm{Ddry}]}$$

$$= \frac{[\mathrm{MACOEFdry}]\,[\mathrm{Ddry}] + [\mathrm{MACOEFh2o}]\,[\mathrm{D'h2o}]}{[\mathrm{MACOEFdry}]\,[\mathrm{Ddry}]}$$

where D'h2o = partial H_2O density in the sample = [H2Ofract] [Ddry]. Thus,

$$\frac{[\mathrm{CRdry}]}{[\mathrm{CRwet}]} = \frac{[\mathrm{MACOEFdry}]\,[\mathrm{Ddry}] + [\mathrm{MACOEFh2o}]\,[\mathrm{H2Ofract}]\,[\mathrm{Ddry}]}{[\mathrm{MACOEFdry}]\,[\mathrm{Ddry}]}$$

$$= 1 + [\mathrm{H2Ofract}]\,\frac{[\mathrm{MACOEFh2o}]}{[\mathrm{MACOEFdry}]}$$

This equation is the same as that for the [DRdry]/[DRwet] given by Zimmerman (1971), where DR = dose rate for gammas. If the moisture correction factor is defined as [MACOEFh2o]/[MACOEFdry], then,

$$\frac{[\mathrm{CRdry}]}{[\mathrm{CRwet}]} = 1 + [\mathrm{H2Ofract}]\,[\text{moisture corr. factor}]$$

The attenuation of gammas by water is evident in all three plots (figures 21, 22, and 23). The excessive scatter for U (lower R**2) may be attributed to the plotted as a function of water content (figures 24, 25, and 26) and the concentration ratio is equal to the ratio of the count rates (a simplification), then the moisture correction factor can be evaluated by multiplying the slopes of the fitted lines (abscissa = percent H_2O) by 100 (converts abscissa to fraction H_2O) and the biases between the two methods from their intercepts. However, rather than using the unweighted linear fits shown in the figures, we performed weighted York fits in each case (Ludwig, 1993) and obtained slightly different slopes. Table 11 compares the resulting moisture correction factors to factors computed from the mass absorption coefficients listed in George et al. (1985, p. 43).

Table 11. Comparison between moisture correction factors from this study and factors computed from the mass absorption coefficients listed by George et al. (1985).

	This Study	George et al. (1985)
K (1.46 MeV)	1.46 ± 0.11	1.25
U (1.76 MeV)	0.78 ± 0.17	1.24
Th (2.61 MeV)	1.58 ± 0.16	1.21

Since the values do not vary strongly with gamma energy over this energy range and the uncertainties are relatively large, the same value can be used for all three gamma energies. Also, the values for K and Th would be expected to be more reliable than that for U, because of the effect of radon. Thus, we adopted a value of 1.4 ± 0.1 for the moisture correction factor. This correction factor was then applied to the field gamma spectrometry data and the resulting graphs of moisture corrected field vs. laboratory gamma spectrometry (figures 27, 28, and 29) show much better agreement. In all three cases, the scatter decreased and the slope more nearly approached 1.0. The fitted lines still lie below the 45-degree lines so a value for the moisture correction factor slightly larger than 1.4 may be appropriate. We cannot correct for radon migration and thus, the scatter for U remains larger than that for K or Th.

Procedure for Computing Concentrations of K, U, and Th from Field Gamma Spectrometry.

The raw counts recorded for each window are converted to net counts per second by dividing by the counting time in seconds and subtracting the background cps in each window (for Savannah, Georgia, from Table 7). The 4-pi calibration coefficients (for the most recent date in Table 8) are then used to compute percent K, ppm eU, and ppm eTh. These concentrations are corrected for attenuation of gammas by water by multiplying each concentration by the factor $(1 + 1.4 * \% H_2O / 100.)$. Table 12 shows estimated CV's for each parameter.

Table 12. Estimated coefficient of variation (CV) for each parameter.

Parameter	CV
Moisture correction factor	10 %
H_2O content when sample collected	10 %
H_2O content over period of burial	30 %
Alpha k-effective	30 %
H_2O attenuation for alpha, beta, and gamma	20 %

Computation of Dose Rate

The annual dose rate to the sample, in units of Grays/kyr, is computed from the concentrations of K, U, and Th by the method described in Aitken (Aitken 1985, p. 10-12 and p. 282-288). We assume secular equilibrium in the uranium decay series. We are no longer dealing exclusively with gamma radiation and must now include alpha, beta, and cosmic radiation and factor in the reduced effect of alpha radiation relative to beta and gamma radiation. The annual radiation doses shown in Table 13 are adapted from Aitken's Table 1.1 (1985, p. 12).

Table 13. Annual radiation doses in Grays/kyr (adapted from Aitken, 1985, p.12).

	Alpha	Beta	Gamma
1% Rb		4.	
1% K		0.83	0.24
1 ppm U	2.78	0.147	0.113
1 ppm Th	0.739	0.029	0.051

We compute water attenuation corrections for each type of radiation using the equations:

(H2O corr alpha) = 1 / [1 + 1.50 * (percent H2O)/100]
(H2O corr beta) = 1 / [1 + 1.25 * (percent H2O)/100]
(H2O corr gamma) = 1 / [1 + 1.14 * (percent H2O)/100]

where the moisture correction factors 1.50, 1.25, and 1.14 are taken from Aitken (1985, p. 75). The factor 1.14 originally came from Zimmerman (1971) and, unlike the moisture correction factor computed for gamma spectrometry of K, U, and Th, it is valid for the mix of gamma energies from the various decay chains. Note that Rendell (1985b) recommends using a factor of 1.10 rather than 1.14, while Berger (1988) lists a value of 1.00 for this factor. Aitken and Xie (1990) present a more recent discussion of these factors.

It is known that a given dose of alpha radiation produces a smaller TL signal than does an equal dose of beta or gamma radiation. Aitken's Appendix K (1985, pp.308-317) and Berger (1988) discuss the several systems (a-value, b-value, k-value) which are used to take account of this reduced effectiveness for alphas. Rather than evaluate one of these factors for each sample, we computed an average a-value for a set of nineteen loesses (Forman et al., 1992, twelve Mississippi Valley and Missouri Valley loesses; Forman, 1993, seven Snake River Plain loesses) and converted these to k-effective values by multiplying by the factor 0.84 (Aitken, 1985, p. 313). The value obtained for k-effective is 0.10 ± 0.03. The relatively large standard deviation reflects the range of values found for these loesses.

We now compute the dose rates for element/radiation combinations using the annual radiation doses in Table 13, the corrections for attenuation by water, and the alpha k-effective value (0.10 ± 0.03) according to the following equations:

(dose rate Rb beta) = 4. * (percent K/200.) * (H2O corr beta)

(dose rate K beta) = 0.83 * (percent K) * (H2O corr beta)
(dose rate K gamma) = 0.24 * (percent K) * (H2O corr gamma)

(dose rate U alpha) = 2.78 * (ppm eU) * (H2O corr alpha) * 0.10
(dose rate U beta) = 0.147 * (ppm eU) * (H2O corr beta)
(dose rate U gamma) = 0.113 * (ppm eU) * (H2O corr gamma)

(dose rate Th alpha) = 0.739 * (ppm eTh) * (H2O corr alpha) * 0.10
(dose rate Th beta) = 0.029 * (ppm eTh) * (H2O corr beta)
(dose rate Th gamma) = 0.051 * (ppm eTh) * (H2O corr gamma)

The dose rates for the various element/radiation combinations are combined to give the dose rates for the radioelements as follows:

(dose rate Rb) = (dose rate Rb beta)
(dose rate K) = (dose rate K beta) + (dose rate K gamma)
(dose rate U) = (dose rate U alpha) + (dose rate U beta) + (dose rate U gamma)
(dose rate Th) = (dose rate Th alpha) + (dose rate Th beta) + (dose rate Thgamma)

The value, 0.291 Gy/kyr, for cosmic ray dose at sea level and latitiude 38 degrees south is taken from Prescott and Hutton (1988). This value is valid for latitudes greater than 40 degrees but must be corrected for the sample's elevation above sea level and depth within the sediment. That is:

(dose rate CR) = (0.291 Gy/kyr) * (elev factor) * (depth factor)

Aitken (1985, p. 298) presents a graph of the (elev factor) vs. elevation for three different latitudes. The low elevation portion of each curve (certainly below about 3000 meters or 9800 feet) is approximately linear with slopes shown in Table 14.

Table 14. Rate of change of cosmic ray intensity with elevation at various latitudes (adapted from Aitken, 1985, p.298).

Latitude	Slope
>40 deg	8.2e-5/foot
25 deg	6.1e-5/foot
0 deg	4.0e-5/foot

Thus,

(elev factor) = 1.000 + (slope)*(elevation, feet).

Values for the (depth factor) are taken from the data presented by Prescott and Hutton (1988) and may be read from figure 30, which is computed from these data assuming a density of 2.0 gm/cm3 for the sedimentary material. From 0 to 0.75 meters, values for the dose rate were read from figure 1 of the Prescott and Hutton paper. Over depths from 0.75 to 15 meters, we used their relationship,

dose rate = (0.21 Gy/kyr) * exp[-0.70 x + 0.0005 x 2] ,

where x = depth (in cm) * (2.0 g/cm3) / 100. Finally, the total dose rate seen by the sample is computed:

total dose rate = (dose rate CR) + (dose rate Rb)
+ (dose rate K) + (dose rate U) + (dose rate Th)

Table 15 shows total dose rates computed for our set of samples.

Table 15. Natural radiation dose rates for typical samples (see Table 16 for the type of sediment). Data computed assuming the alpha k-effective = 0.10 ± 0.03 and the moisture content over geologic time equals the water content when the sample was collected (unless indicated otherwise).

Sample	K, %	U, ppm	Th, ppm	Elevation m (feet)	Depth m	H_2O %	Dose Rate Gy/kyr (CV)
Northern Virginia Piedmont:							
NVP-2	1.23 ±0.06	2.97 ±0.41	8.59 ±0.34	98 (320)	0.30	16	3.74 (7.9%)
Wittsburg Quarry, Arkansas:							
MMV-5	1.51 ±0.07	3.63 ±0.43	8.00 ±0.32	119 (390)	9	16	4.07 (8.1%)
MMV-6	1.84 ±0.08	3.67 ±0.45	8.44 ±0.36	"	5	22	4.22 (7.9%)
MMV-7	1.41 ±0.07	3.30 ±0.50	10.86 ±0.44	"	7	19	4.06 (8.4%)
MMV-9	1.84 ±0.07	2.95 ±0.42	8.63 ±0.34	"	10	17	4.11 (7.4%)
Phillips Bayou, Arkansas:							
MMV-18	1.54 ±0.07	3.47 ±0.40	6.75 ±0.31	64 (210)	4.2	21 (80% of saturation)	3.72 (8.0%)
MMV-19	1.85 ±0.08	4.08 ±0.48	8.61 ±0.36	"	5.8	21	4.44 (8.0%)
MMV-20	1.86 ±0.08	3.93 ±0.47	8.58 ±0.36	"	7.8	21	4.36 (8.0%)
MMV-21	1.16 ±0.05	3.05 ±0.45	9.72 ±0.35	"	9.4	9	3.99 (8.3%)
MMV-22	1.91 ±0.08	3.32 ±0.43	8.53 ±0.36	"	12.9	21	4.12 (7.7%)
Loveland Paratype Section, Iowa:							
MVL-3	1.58 ±0.07	3.93 ±0.49	9.71 ±0.34	335 (1100)	32	7	4.90 (8.1%)
MVL-4	1.65 ±0.07	3.76 ±0.46	9.03 ±0.32	"	41	7	4.82 (7.9%)
MVL-2	1.41 ±0.06	3.48 ±0.42	7.91 ±0.31	"	44	14	3.94 (8.2%)
Northeastern Colorado:							
NEC-18	3.12 ±0.10	1.96 ±0.34	7.74 ±0.28	1494 (4900)	0.85	5	5.58 (4.8%)
NEC-17	3.12 ±0.10	2.02 ±0.34	7.84 ±0.28	"	1.12	5	5.60 (4.9%)
NEC-20	2.74 ±0.09	3.25 ±0.51	11.43 ±0.39	1524 (5000)	0.70	5	6.35 (6.2%)
NEC-19	2.80 ±0.10	3.66 ±0.57	12.88 ±0.45	"	1.00	7	6.65 (6.6%)

Table 15. (continued)

Sample	K, %	U, ppm	Th, ppm	Elevation m (feet)	Depth m	H_2O %	Dose Rate Gy/kyr (CV)
Yucca Valley, California:							
YVC-1	2.61 ±0.10	3.81 ±0.62	14.46 ±0.48	910 (3000)	2	3	7.04 (7.0%)
YVC-2	2.58 ±0.09	3.79 ±0.62	14.44 ±0.48	"	2	3	6.95 (7.0%)
YVC-8				"	0	0	~7 (based on YVC-1 and 2)
Marryat Creek, Australia:							
MCS-3a	1.84 ±0.01	1.06 ±0.22	8.22 ±0.27	500 (1640)	0.15	2	4.05 (5.2%)
MCS-3b	1.49 ±0.01	1.13 ±0.08	7.63 ±0.29	"	0.40	2	3.56 (5.6%)

Computation of Age

As stated in the Introduction, TL ages are calculated by dividing the equivalent dose by the annual dose rate. The TL ages computed for the set of samples in Table 15 are listed in Table 16. Of the 21 ages, two (MMV-7 and MVL-3) appear to be too old. There is no indication from the raw data that these should be suspect. MMV-7 may have been affected by intense pedogenesis, although the age for the other Sangamon sample, MMV-21, appears valid. Our age for MVL-3 is clearly too old for Peoria loess. These 21 samples were the first ones run by this procedure and illustrate the necessity for sampling within a good stratigraphic framework in order to be able to spot problems later. The other 19 TL ages in Table 16 are consistent with the stratigraphy and agree quite well with other age determinations.

Schedule

Table 17 shows values for the parameters which can be used to establish an efficient schedule for processing samples. One strategy which may be followed is:

1) recognition that the rate determining operation is the beta irradiation (except for young samples).
2) starting one set of samples each month where the sum of ages = 180 Kyr (i.e., the cycle time for the beta irradiator = 28 days). For example, Peoria + Roxana + Sangamon or Loveland.
3) the number of samples in each set is determined by the distribution of ages among the samples.

This approach does not take into account data points which may need to be added after the growth curve has been constructed.

Table 16. TL ages for typical samples. Weighted averages are computed with program ISOPLOT (Ludwig, 1993) ignoring "external error". These ages may be compared to the "reasonable ranges of time for deposition of the loess" based on a variety of ages in the literature and data for Be-10 (Pavich, 1993) or to the specifically referenced "other age determinations". Ages which do not compare well (considering the statistics) are indicated with a "**".

TB = total bleach method
PB = partial bleach method.

Conditions:

Sunlight bleach time, TB = 16 hours, except:
16.2 hours for MMV-5
16.3 hours for NEC-19 and 20

Sunlight bleach time, PB = 1 hour

Preheat = 64 hr at 124°C, except:
44 hr at 124°C for MMV-5, 6, 7, and 9 and
48 hr at 124°C for NEC-17, 18, 19, and 20

Optical filters = BG-39, 7-59, Pyrex except:
UG-11, CS1-75 for MMV- 5, 6, 7, and 9
BG-39, 7-59 for NEC-17, 18, 19, and 20

Ramprate = 5°C/sec, except:
10°C/sec for MMV-5, 6, 7, and 9

Reasonable ranges of time for deposition of loess:

Peoria Loess	25	-	9	kyr BP
Roxana Silt/Pisgah Fm. (Farmdale and unnamed soils)	65	-	26	
Loveland Loess (Sangamon soil)	190(?)	-	120(?)	

Sample	Stratigraphic Unit	Equivalent Dose Method	Plateau Region, °C	Equivalent Dose, Gy	TL Age, kyr	Other Age Determinations, kyr
Northern Virginia Piedmont:						
NVP-2	loess	TB	350-370	50.6 (3%)	13.5 ± 1.1	
		PB	380-450	63.7 (19%)	17.0 ± 3.5	
				weighted average =	13.8 ± 1.0	

Sample	Stratigraphic Unit	Equivalent Dose Method	Plateau Region, °C	Equivalent Dose, Gy	TL Age, kyr	Other Age Determinations, kyr
Wittsburg Quarry, Arkansas:						
MMV-5	lower Peoria loess	TB	330-350	94.3 (5.6%)	23.2 ± 2.3	
MMV-6	Roxana silt	TB	380-410	142.9 (3.1%)	33.9 ± 2.9	
MMV-7	Sangamon	TB	390-430	970 (12%)	240 ± 40	**
MMV-9	Loveland loess	TB	410-430	790 (16%)	190 ± 30	
Phillips Bayou, Arkansas:						
MMV-18	lower Peoria loess	TB	360-400	97.7 (3.3%)	26.3 ± 2.3	
		PB	380-400	76.5 (16%)	20.6 ± 3.7	
				weighted average =	24.7 ± 1.9	
MMV-19	upper Roxana silt	TB	360-410	139.1 (2.7%)	31.3 ± 2.6	
		PB	350-390	172.1 (5.7%)	38.8 ± 3.8	
				weighted average =	± 33.7 ± 2.1	44 (Be-10, Pavich, 1993)
MMV-20	lower Roxana silt	TB	350-420	190.5 (3.8%)	43.7 ± 3.9	
		PB	350-430	218.6 (6.0%)	50.1 ± 5.0	
				weighted average =	46.1 ± 3.0	60 (Be-10, Pavich, 1993)
MMV-21	top Sangamon	TB	310-390	481 (10%)	121 ± 16	
		PB	350-390	489 (20%)	123 ± 27	
				weighted average =	122 ± 14	124 (Be-10, Pavich, 1993)
MMV-22	Loveland loess	TB	430-450	798 (19%)	194 ± 40	
		PB	420-440	715 (24%)	174 ± 43	
					184 ± 29	183 (Be-10, Pavich, 1993)

Sample	Stratigraphic Unit	Equivalent Dose Method	Plateau Region, °C	Equivalent Dose, Gy	TL Age, kyr	Other Age Determinations, kyr
Loveland Paratype Section, Iowa:						
MVL-3	middle Peoria loess	TB	390-430	207.8 (6.3%)	42.4 ± 4.4	
		PB	370-430	176.9 (12%)	36.1 ± 5.2	
				weighted average =	39.8 ± 3.3	** 21 ± 3 (TL, Forman et al., 1992)
MVL-4	lower Peoria loess	TB	380-430	122.7 (6.8%)	25.5 ± 2.7	
		PB	370-430	101.6 (23%)	25.8 ± 6.3	
				weighted average =	25.5 ± 2.5	18 ± 2 (TL, Forman et al., 1992)
MVL-2	lower Pisgah Fm.	TB	360-420	148.8 (11%)	37.8 ± 5.1	
Northeastern Colorado:						
NEC-20	eolian sand, middle Qe3 deposit	TB	320-370	24.7 (4%)	3.9 ± 0.3	
NEC-18	eolian sand, bottom Qe3 deposit	TB	330-370	26.0 (14%)	4.7 ± 0.7	
		PB	300-350	30.5 (16%)	5.5 ± 0.9	
				weighted average =	5.0 ± 0.6	
NEC-19	eolian sand, bottom Qe3 deposit	TB	350-380	40.9 (4%)	6.1 ± 0.5	
		PB	340-370	35.4 (12%)	5.3 ± 0.7	
				weighted average =	5.9 ± 0.4	
	top Qe2 deposit					7.3 ± 0.1 (RC) 8.8 ± 1.7 (TL) (Forman and Maat, 1990)
NEC-17	eolian sand, bottom Qe2 deposit	TB	310-350	51.7 (6.6%)	9.2 ± 0.8	
		PB	320-350	43.4 (21%)	7.7 ± 1.7	
				weighted average =	9.0 ± 0.7	
	top Qe1 deposit					10.5 ± 0.4(RC, Forman, Goetz, and Yuhas,1992)

Sample	Stratigraphic Unit	Equivalent Dose Method	Plateau Region, °C	Equivalent Dose, Gy	TL Age, kyr	Other Age Determinations, kyr
Yucca Valley, California:						
						8.1 +0.4/-0.2 (just above YVC-1, Hecker and Schwartz, 1993)
YVC-1	upper silt layer	TB	330-370	60.1 (10%)	8.5 ± 1.0	
		PB	330-360	59.8 (16%)	8.5 ± 1.4	
				weighted average =	8.5 ± 0.8	
YVC-2	lower silt layer	TB	370-400	85.0 (5.5%)	12.2 ± 1.1	
		PB	380-400	94.9 (15%)	13.7 ± 2.3	
				weighted average =	12.5 ± 1.0	
YVC-8	surface silt	TB	290-320	7.4 (16%)	~1.	
Marryat Creek, Australia:						
MCS-3a	eolian sand	TB	340-370	50.9 (7%)	12.6 ± 1.1	
		PB	360-390	49.2 (15%)	12.1 ± 1.9	
				weighted average =	12.5 ± 1.0	Holocene (soil texture, Machette and Crone, 1993, p. B13)
MCS-3b	colluvial/ fluvial gravel	TB	360-390	138 (4%)	38.8 ± 2.7	
		PB	370-410	144 (7.4%)	40.4 ± 3.7	
				weighted average =	39.4 ± 2.2	52-131 ($CaCO_3$ accumulation, Machette and Crone, 1993, p.B24)

Table 17. Parameters for TL schedule.

Computation of Beta irradiation times:

Assumptions: alpha K-effective = 0.15
1.4 %K x 1.1 Gy/kyr/%K = 1.5 Gy/kyr
2.5 ppmU x 0.7 Gy/kyr/ppmU = 1.7 Gy/kyr
7.5 ppmTh x 0.2 Gy/kyr/ppmTh = 1.5 Gy/kyr

Natural Dose Rate = $\overline{\sim 5}$ Gy/kyr

ED (equivalent dose) = (~5 Gy/kyr) x (Age,kyr)

distribution of data points for growth curve =

0 x ED (N)
(0.5 x ED)
1 x ED
2 x ED
4 x ED
8 x ED

Beta Irradiator Dose Rate = 3 Gy/min

Beta Irradiation Time (days) per sample =

$$\frac{\text{(ED)}}{\text{(Beta Irradiator Dose Rate)}} \times (1 + 2 + 4 + 8) \times \text{(4 disks)} \times \text{(2 experiments)}$$

$$= \frac{(\sim 5\ \text{Gy/kyr}) \times (\text{Age,kyr})}{(3\ \text{Gy/min})} \times 120 \times \frac{1}{1440\ \text{min/day}}$$

$$= (0.138) \times (\text{Age,kyr})$$

Age		ED	Beta Irradiation Time / Sample	Sample Throughput
10 kyr		50 Gy	1.4 days/sample	260 samples/year
20	(Peoria)	100	2.8	130
40	(Roxana)	200	5.5	66
60		300	8.3	44
80		400	11.0	33
100		500	13.8	26
120		600	16.6	22
140	(Loveland)	700	19.3	19
160		800	22.1	17
180		900	24.8	15

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Appendix A. Sample Collection Procedures

A.1. Equipment Needed

35-mm camera
black, opaque cloth (45" x 60")
field notebook
tape measure (cm)
Notebook PC
portable gamma spectrometer with 3"x3" NaI gamma detector
sample tubes (2" inside diameter by 6" long, 0.15" wall PVC tube, bevelled at ends, see figure 31)
black, opaque caps for sample tubes (Caplugs, Clover Industries)
black polyethylene film bags
drive tube (steel, 1-foot long, see figure 32)
2-pound sledge hammer
35-mm photo film canisters
1-quart, 2.7-mil polyethylene freezer bags
roll of black, vinyl 3/4"-wide electrical tape
4-inch auger with 2-foot extension rod and handle
cover plate for back of 4-inch auger (for vertical augering)
2 adjustable wrenches (to remove extension rod and handle)
knife
trowel

A.2. Sampling Procedure for Horizontal Hole in Vertical Exposures (Primarily Loess)

choose position free of cracks, roots, krotovina, yellow streaks (due to oxidation), carbonate, and pebbles or stones (desert environments)
Note: if the only position available is one in which cracks, roots, or krotovina may be encountered at depth or the sediment is so hard that it will be very difficult to remove a sample tube driven in at depth, then take sample at the surface of the exposure
ideally, position is at least 0.5 meter above or below boundary of layer so gamma counting is performed in a homogeneous volume
auger sample hole 1 foot into face of exposure
put black cap on back of sample tube
place capped end of sample tube on drive tube and pound into back of sample hole with sledge hammer as far as the cap
remove drive tube
free up the buried sample tube by digging around it with the knife (this may take a long time if the sediment is hard due to the presence of carbonate), and break free (without pulling it out) by pushing the trowel alongside it and levering it sideways
cover yourself and the sample hole with the black, opaque cloth (this may require the aid of others) and remove the sample tube from the hole and quickly cover the open end with a black cap
seal both caps to the sample tube with black vinyl electrical tape
write the sample number on the tube with a Sharpie
place the sealed sample tube in a black polyethylene film bag, roll up, and seal with a label
write the sample number and date on the label with a Sharpie

A.3. Sampling Procedure for Vertical Hole in Surface Locality (Primarily Sand)

auger down to get into C horizon (may be 1 or more meters deep)
cover hole with black cloth
place cover plate on back of auger bucket
place auger in hole with black cloth around extension pipe
fill auger bucket by augering bottom of hole
withdraw auger bucket (keep it covered with black cloth)
reach under black cloth, remove cover plate from back of bucket, and scrape off top 0.5 inch of sand
place sample tube (with cap on rear) in back of bucket between yoke and push pipe about 3 inches into sample
remove black cloth and pound sample tube in the rest of the way
free the sample tube by digging around the side with fingers and by inserting trowel along the side
cover again with the black cloth, lift the auger handle, and remove the sample tube thru the bottom of the yoke
place black cap over open end of sample tube
seal and label as above

A.4. Collection of Bulk Sample for Determination of Moisture Content, Laboratory Gamma Counting, and Delayed Neutron Counting

auger back in the sample hole, filling the auger bucket with sediment
transfer the sediment to a freezer bag with the trowel, filling the bag about 3/4 full
close the bag and write the sample number and date on the label area
insert sample bag in a second freezer bag and close (the first bag containing the sample will just slide into the second bag if the correct amount of sample has been collected)
store the bulk sample away from from direct sunlight (to avoid distilling water from the sample)

A.5. Information to be Recorded

sample number (and date)
stratigraphy
location (quad, county, state, distances, position in stratigraphic section, depth below original surface)
description
counting (depth of insertion of detector, geometry, counting time, and counts in K, U, Th, and CR windows)
sampling (e.g., sample tube + freezer bag)
get a description from the geologist of the sample and note the presence or absence of cracks, roots, and krotovina

A.6. In Situ Gamma Counting

auger the sample hole to a depth of 2 feet
use the auger (and the trowel if necessary) to enlarge the hole to a diameter which will accept the NaI gamma detector
insert the NaI gamma detector to a depth of 20 inches (the rear end of the detector is 7 inches into the hole)
collect two 999-second counts, storing each spectrum in memory (see Appendix B)

A.7. Efficient Sampling

about one sample can be collected per hour if everything goes smoothly
the next sample position should be determined, augered, and sampled while the previous one is counting
nails with colored disks should be placed at layer and horizon boundaries during sampling to be available for later photography
photographs can be taken faster after sampling is completed (usually one at distance to locate the hole in stratigraphic context and one close up to show the texture of the sediment at the

position of the sample); the augered holes often show up in distant photographs of the site

sketch the stratigraphy roughly during sampling (indicating items that will show up in photographs and the locations from which other dated samples were collected) and then place positions on a more detailed stratigraphic diagram later

Appendix B. Procedures for Operating Exploranium Portable Gamma Spectrometer

Manuals:
- Portable Gamma Ray Spectrometer Manual
- Geological Applications Manual

To turn on and off:

ON/OFF to ON   gives MONITOR condition (8 hyphens, MON)

Special Keys:

START/STOP - start measurement or stop any activity
CLEAR - clear data loaded into display and erase contents of data memory
STORE - store parameters or measured data in memory
SHIFT - change to red functions in upper part of keyboard
up arrow, down arrow - increment or decrement display thru current function

To check batteries:

batteries are checked every 20 sec:
< 6.5 V - gives <BAT> (batteries good for 1 more hour)
< 5.5 V - gives error E2 (must change batteries)

or, SHIFT, USER, 2 - displays voltage at 1 sec intervals
START/STOP (1 sec) - terminates test

To set or read live time:

SHIFT, TIME, enter time (1-999 sec), STORE
START/STOP (1 sec)

To set or read ROI channel ranges:

SHIFT, ROI - gives <SET> and window number, L or H
up arrow, down arrow - advances thru windows
enter channel number, STORE
START/STOP (1 sec) - terminates entry, starts test

Note: Window
1 - TC (ch= 70-255)
2 - K (ch=112-126, 1.46 MeV from K-40)
3 - U (ch=134-152, 1.76 MeV from Bi-214)
4 - Th (ch=199-221, 2.62 MeV from Tl-208)
5 - CR (ch=222-255)

To set display mode:

SHIFT, C/C - toggles between Count display and Concentration display, <CONC>
START/STOP (1 sec) - terminates entry

Note: overflow in 511-keV peak and other low energy channels makes Total Counts meaningless most of the time

To display channel data:

SHIFT, SPECTRUM, 0
up arrow, down arrow - advances thru channels

To position detector for 4-pi gamma counting:

```
/__________ ____________
   Hole    |  Detector  |
           |            |
___________|____________|
/
<--- 7" ---> <--- 13" ---->
<---------- 20" --------->
```

To erase previous spectrum and begin accumulation of new spectrum:
START/STOP (1 sec) - gives <STAB> for 4 sec, then <STAB>, <ACC>, remaining LT decrements
at end of count - spectrum is processed and Total Count or PPM eU displays
up arrow, down arrow - advances thru windows
START/STOP - terminate accumulation manually

To reprocess spectrum with new ROI windows or new display mode:
SHIFT, SPECTRUM, 2 - spectrum is reprocessed

To initiate memory:
SHIFT, TIME - check that LT is set, all spectra stored later must have this LT
SHIFT, HEADER - gives first 8 bytes of memory

(START/STOP to exit before destroying data in current memory)

CLEAR, CLEAR, CLEAR - runs Memory Test (40 sec), then gives H
enter date (mmddyy)
STORE, STORE, STORE

To transfer spectrum from working window to memory:
SHIFT, USER, 6, N, STORE, where N = spectrum number (1-62)
- gives <MON> after spectrum stored

To transfer spectrum from memory to working window:
SHIFT, USER, 3, N, STORE, where N = spectrum number (1-62)

Error: E4 = spectrum is empty

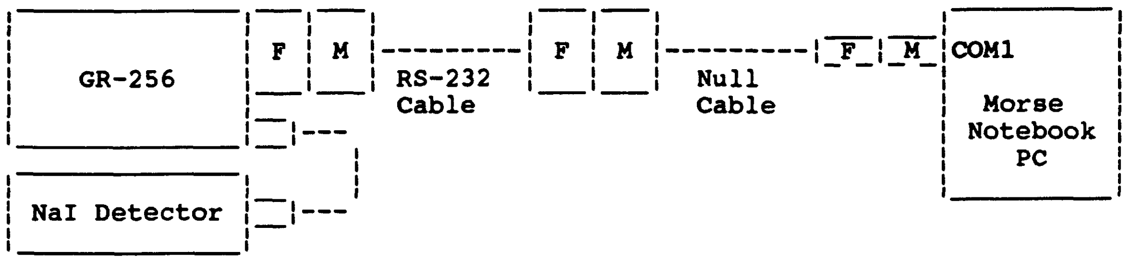
To set baud rate:
SHIFT, USER, 8
up arrow - steps thru baud rates (use 9600)
STORE

To run and stop GWBASIC programs on Morse Notebook:

```
> CD \EXPLORAN
> GWBASIC
(note: BASIC program must open COM1:
      OPEN "COM1:1200,N,8,1,CS0,DS0,CD0" as #idevcom1
      OPEN "COM1:9600,N,8,1,RS,DS65535" as #1 )
F3 (Load), filename.BAS
F1 (List)
F2 (Run)
```

ESC or CTRL/BREAK - stops program
type SYSTEM - returns to DOS

Cabling between GR-256 and Morse Notebook:



To test communications between GR-256 and Morse Notebook PC:

on GR-256: SHIFT, TIME, set time = 10 sec, STORE
SHIFT, REMOTE

on Notebook: > GWBASIC
F3 (Load), type DEMO1.BAS
F2 (Run)
RETURN

cycles thru:

1) spectrum accumulation
2) spectrum transfer to Notebook PC
3) spectrum decoding
4) spectrum display (can print spectrum)

Note: NaI detector must be connected to GR-256 or only 1, 2, and 3 take place.
Note: "?" sent from Notebook PC to GR-256 causes contents of GR-256 display to be transferred to Notebook PC.
Errors in DEMO1.BAS:
Device Fault in 2480 - printer not connected

To take GR-256 out of REMOTE:
START/STOP 1 sec
Note: Notebook PC must not be addressing GR-256.

To transfer one spectrum from GR-256 memory to Notebook PC and store as ASCII file:

on GR-256: SHIFT, USER, 3, N, STORE, where N = spectrum number (1-62)
- transfer spectrum from memory to working window
SHIFT, USER, 8 - set baud rate
SHIFT, REMOTE - place in remote

on Notebook PC: > GWBASIC
F3 (Load), enter "SPE257.BAS
F2 (Run)
RETURN
DO YOU WISH TO SAVE A SPECTRUM TO DISK? Y
ENTER FILENAME? SPEnn.DAT

```
 ---> on GR-256:        START/STOP
|                       SHIFT, USER, 3, N, STORE
|                       SHIFT, REMOTE
|
|    on Notebook PC:    F2
|                       RETURN
|                       DO YOU WISH TO SAVE A SPECTRUM TO DISK?   Y
|                       ENTER FILENAME?   SPEnn.DAT
|_________________________________|
```

To transfer range of spectra in GR-256 memory to Notebook and store as ASCII files (GR2NB.ASC format):

```
on GR-256:        SHIFT, TIME, set time to value for spectra, STORE
                  SHIFT, USER, 8           - set baud rate
                  SHIFT, REMOTE            - place in remote

on Notebook:      > GWBASIC
                  F3 (Load), enter "GR2NB.ASC
                  F2 (Run)
                  enter range of spectra
                  RETURN
```

Spectra are transferred to files SPE01.DAT ... SPEnn.DAT.

To reformat spectra from GR2NB.ASC format to .SPE format on Notebook PC:

```
> RUN UTILITY
enter 2, range of spectra (Note: spectra must be in same directory as
                          UTILITY)
```

Reformatted spectra are stored in files SPE01.SPE ... SPEnn.SPE.

To display and plot ASCII spectra in .DAT files on Notebook PC:

```
> GWBASIC
F3 (Load), enter "R257LIN.BAS
           or, "R257LOG.BAS
           or, "SPEHC.BAS
F2 (Run)
enter SPEnn.dat
RETURN
```

Note: INT = maximum channel count rather than sum of counts.

Appendix C. Procedures for Treating Bulk Sample

C.1. Determination of Moisture Content

weigh while wet in inner freezer bag
spread sample out on paper to air dry
disaggregate clumps by hand
allow to air dry for several days (occasionally repeat disaggregation)
return to freezer bag and weigh dry sample
subtract mass of freezer bag (about 7 g) from each weighing and compute % H2O:

% H2O =
100 x [(mass wet sample) - (mass dry sample)] / (mass dry sample)

C.2. Crushing in Jaw Crusher

if air drying yields large, very hard clumps, pass sample through jaw crusher with ceramic plates reserved for soft sediment samples
sieve with No. 10 sieve (2 mm) and repeat crushing with retained material

C.3. Determination of Saturation Moisture Content

place about 15 g. dried sample in a 40-ml plastic centrifuge tube
weigh dry sample + centrifuge tube
add water and stir until water stands above sample
allow to settle for 1 hour
centrifuge (setting = 7) for 5.0 minutes
remove water above sample with aspirator (ignore clay in suspension)
weigh wet sample + centrifuge tube
subtract mass of centrifuge tube (about 12 g) from each weighing and compute saturation moisture content:

% saturation H2O =
100 x [(mass wet sample) - (mass dry sample)] / (mass dry sample)

C.4. Laboratory Gamma Spectrometry to Determine K, U, and Th (Bunker and Bush, 1966; Bunker and Bush, 1967)

To prepare samples for counting:
weigh 600.0 +- 0.5 grams into 6"-diameter x 1.5"-high plastic canister
seal top on with 2 layers of black vinyl electrical tape
label with masking tape (record lab number, weight, and date)

To determine bulk density:
place canister on side and shake to settle powder
use vibrator to remove any air pockets
hold special template vertically across middle of canister and read weight of water which corresponds to the volume of sample. Compute the bulk density using the equation:

D = (600.0 g) / (Weight of water)

(e.g., 600.0 / 520 = 1.15 g/cm^3)

record bulk density on label

Counting and computing equipment:
4" x 5" NaI (Tl) scintillation detectors
Canberra AccuSpec MCA PC card
MCA PC
data reduction PC

To turn on HV to counters:
HV = +1000 V
allow to stabilize 1 day before counting

Load samples and fill in Sample Log Sheet

To start and operate MCA program on PC:

```
MCA> MCA
Acquire, Preset, Live, enter live time (seconds)

ESC          - backup one menu level
\            - return to Main Menu
F10          - advance ADC number
F9           - backup ADC number
SHIFT/F2     - erase spectrum
F1           - acquire on/off toggle
Exit,Y       - exit MCA program
```

To read out spectra to disk file FOR001.DAT:

```
MCA> OUTPUT
enter Detector, Sample Name, File Number, Weight, Density, etc.
      from Sample Log Sheet for each detector
      (terminate with null Detector)
```

To examine spectral files in disk file FOR001.DAT:

```
MCA> BROWSE FOR001.DAT
Page Up, Page Down, Arrow keys      (move thru file)
CTRL/C                              (exit BROWSE)
```

To migrate disk file FOR001.DAT from MCA PC to 3.5" disk:

```
MCA> COPY FOR001.DAT A:*.*          (copy file to 3.5" disk)
MCA> DEL FOR001.DAT                 (removes current disk
                                     file so next version
                                     can be initialized)
```

To migrate disk file FOR001.DAT from 3.5" disk to data reduction PC:

```
> CD \GAMMA
GAMMA> COPY A:FOR001.DAT *.*        (copy file to data
                                     reduction PC)
GAMMA> COPY FOR001.DAT FOR051.DAT   (input file to GAM01)
GAMMA> DEL FOR061.DAT               (output file from GAM01)
```

To remove last line from each spectral file:

```
GAMMA> WP                           (starts Word Perfect)

CTRL/F5                - gives Text In Out Menu
1                      - DOS Text
2                      - Retrieve
enter FOR051.DAT

ALT/E                  - macro which edits out last line of
                              each spectrum
HOME,HOME,down arrow   - go to end of file
CTRL/E                 - deletes last line in file
```

```
CTRL/F5                    - gives Text In Out Menu
2                          - Text
1                          - Save
CR                         - Save to Old File
Y                          - Yes, Replace existing file

F7                         - Exit WP
N                          - No, Don't save document
Y                          - Exit WP
```

To run data reduction program:

```
GAMMA> GAM01               ( 5 min/50 spectra)
```

To print out report:

on printer, select "Condensed Print"
on printer switch box, select B

```
GAMMA> PRINT FOR061.DAT      (prints detailed report
                              with summary by detector
                              and sample at end)
```

To transfer ASCII data file to VAX and average values for each element in each sample:

migrate file FOR061.DAT to TL PC
edit out detailed report and leave summary at end
transfer file to VAX with COTERM VAXCABLE
run program GCCALC on VAX

C.5. Delayed Neutron Counting to Determine U and Th (Millard and Keaton, 1982; McKown and Millard, 1987)

To prepare samples:

weigh samples into tared 2-dram Polyvials
label each Polyvial with a Sharpie
heat seal Polyvials with a soldering iron
organize samples into sets of 20
load samples into polyethylene rabbits and each set into a sample magazine
include one oxygen standard (SO), one uranium standard (SU), one thorium standard (ST), and one quality assurance standard (GPP) in each magazine

To prepare sample file from terminal on VAX:

```
$ RUN SYS$DN:SFPREP
Function = DN                  (will give .DAT file)
Output Sample file = DN____
Input Job File     = TTY
     LABNO  FIELDNO  WEIGHT  JOB  SUBMITTER  CHARGE
enter (e.g.,
     ALG-1           12.3456 HM01 MILLARD    4007   ,
     JOB and SUBMITTER only on 1st line of file)
     ...
     CR

Input Job File     = CR
Output Sample File = CR

Function = WT                  (will give .FIL file)
Sample File  = DN____
Do You Want To Create An INAA Sample File?  NO
No. of Samples (F2.0) = n+3
```

```
      give CR's

      delete .DAT file
```

To create directory on VAX for this run and copy sample files to it:

```
            PC                          VAX

      $ VTERM VAXCABLE

                              Username: MILLARD (log onto VAX)
                              Password:
                              $ CWD [.DN]
                              $ CREATE/DIR [.DNnn]
                              $ CWD [.DNnn]
                              $ COPY [-]STAND.DAT *.*
                              $ COPY [-]filename.FIL filename.dat
                              (transfer sample files to new directory)
```

To create and prepare RF.DAT, IF.DAT, RSLTF.DAT, MF.DAT files on VAX:

```
                              $ RUN SYS$DN:DNOPEN
                              $ RUN SYS$DN:DNPREP
                              enter sample filenames (no .DAT)
                              enter run file numbers
```

To copy sample files from VAX to PC:

```
                              SHIFT/F3    (starts KERMIT)

      PC KERMIT> GET filename.DAT
            ...
      SHIFT/F4    (stops KERMIT)

                              $ LO        (logout from VAX)

      CTRL/BREAK
```

To create and prepare RF.DAT, IF.DAT, RSLTF.DAT, MF.DAT files on PC:

```
      $ DNOPEN
      $ DNPREP
      enter sample filenames (no .DAT)
      enter run file numbers
```

To make DN run:

```
      place sample magazine on Changer Block
      attach empty magazine to Discard Block
      have Reactor Operator start PCCTDPC program on CTD
      notify Reactor Operator of the number of samples in the set

      $ @DN

      (Note: DN program now starts)
      choose Debug level (0), Screen display (0), Printer device (1)
      Start at: 1 (Input initial data)
            enter Irradiation, Power, Counter configuration
      Start at: 2 (Input data for next series of samples)
            enter Run File numbers
      Start at: 3 (Start at beginning of next series of samples)

      (Note: DNRUN program now starts)
      Printout should match contents of file DNDATA.DAT.
```

To transfer file DNDATA.DAT from PC to VAX and sort data blocks from DNDATA.DAT to DNSORT.DAT:

```
          PC                          VAX

     $ VTERM VAXCABLE

                               Username: MILLARD (log onto VAX)
                               Password:
                               $ CWD [.DN.DNnn]
                               SHIFT/F3    (starts KERMIT)

     PC KERMIT> SEND DNDATA.DAT
          ...
     SHIFT/F4    (stops KERMIT)

                               $ RUN SYS$DN:DNSORT
                               $ LO        (logout from VAX)
     CTRL/BREAK  (exit VTERM)
```

To process data and generate lab reports:

```
                               $ RUN SYS$DN:DNAUTO
                                  2 = IB (Index blocks for
                                         counter AB)
                               $ COPY IF.DAT IFAB.DAT
                               $ COPY MF.DAT MFAB.DAT
                               $ COPY RSLTF.DAT RSLTFAB.DAT

                                  2 = IB (Index blocks for
                                         counter CD)
                               $ COPY IF.DAT IFCD.DAT
                               $ COPY MF.DAT MFCD.DAT
                               $ COPY RSLTF.DAT RSLTFCD.DAT

                               $ RUN SYS$DN:DNCALC
                                 11 = CF (Combine index and
                                         result files)
                                 12 = LR (Print lab report)
                                 13 = BR (Generate lab report
                                         disk file for BGC)
```

Appendix D. Record Keeping and Flow of Data

D.1. TL Checklist

provides an overall picture of sample status
record date when each step is completed

D.2. Sample Folder

created when a sample starts through the procedure
eventually contains the TL Disks Sheets and all data printouts.

D.3. TL Disks Sheets

four sheets with 6 sets of four disks per sheet
each sheet corresponds to one sample box of 24 disks
disks are grouped by experiment as much as possible (AF, SL, TB, PB)
the treatment given each disk is recorded on the TL Disks sheet for that sample and on the white label on top of the cell well box for that disk (ordinarily done for groups of four disks)
the treatment operations for each set of four disks may be recorded ahead of time; the date when the operation is performed is recorded when completed, e.g.,

```
N + 2h B,  1h SL, Preheat (64 h)
 5/26/92  6/10/92   6/15-18/92
```

enter the Date of IRSL normalization
for the TL glow, enter the Filter Configuration, Run File, Date, NRUN for each disk in the Run File, Experiment File, and NRUN for each disk in the Experiment File (there may be no Run File)

D.4. Clipboards

communicates to others what has been completed and, for example, allows the continuation of operations over weekends by others

Pretreatment:
- allows computation of yields to various fractions
- provides outline of procedure

Pretreatment Work Sheet -
- up to six samples recorded across the top
- check off each operation and record weights

Silt Separation Work Sheet -
- continuation of Pretreatment Work Sheet
- check off each operation and record all information

Sunlight Bleaching

Beta Irradiation

Preheat Oven

D.5. Computer Files

naming:

- all file designations are of the form filename.PRG, filename.DAT, or filename.SUB
- all sample specific filenames and directory names begin with a unique 4 or 5 letter sample designation (e.g., MMV5) for ease in wildcard copying (e.g., >copy MMV5*.* ...)
- all experiment filenames end with an experiment designation (e.g., MMV5TB)
- when it is necessary to create several run files and later merge these into an experiment file, add a letter to the experiment designation (e.g., MMV5TBF.DAT)

.PRG, .DAT, and .SUB files are created before and during the IRSL normalization run (e.g., MMV5NRM1.DAT and MMV5NRM2.DAT)

.PRG program file created in directory \DAYBREAK\PROGRAMS before TL glow run (e.g., MMV5TB.PRG)

.DAT and .SUB data files created in directory \DAYBREAK\TLDATA at time of TL glow run (e.g., MMV5TB.DAT and MMV5TB.SUB)

immediately after glow run:

1) the .DAT and .SUB run files are copied to the sample directory (e.g., \DAYBREAK\MMV5), which contains virgin copies of these files as backups,
2) the .DAT and .SUB files are copied to the sample directory on a 3.5" disk (e.g., B:\MMV5, two samples per disk) - the disk always contains the most recent copy of these files thruout data reduction,
3) delete the .DAT and .SUB files from \DAYBREAK\TLDATA (this can be done later after the other copies have been validated, but should be done on a regular basis to reduce the number of files in \DAYBREAK\TLDATA)

D.6. Data Reduction Printouts

for ease of scanning and recovering data, the printouts for each experiment should be ordered, taped together, and labelled as follows (e.g., see MMV5TB.DAT printout):

- Program (may not be present)
- TAKEDATA Printout (may not be present)
- Synopsis of File
- raw data (several separate plots of glowcurve + background)
- TL Intensity vs Temperature plots:
  - background subtracted (all subtracted glowcurves on one plot)
  - background subtracted, T shifted (all subtracted glowcurves on one plot)
  - background subtracted, T shifted, normalized (all subtracted glowcurves on one plot with run numbers connected to curves)

the data printout after here is specific to each experiment

- for AF:
  - Fade Test plots of Ratio vs Temperature for the several combinations of Stored/Immediate glow, with and without Preheat

for SL:

- Fade Test plots and printouts of N,SL Bleach Time/N Intensity Ratio vs Temperature for each Bleach Time - the ratios at 200°, 250°, 300°, 350°, and 400°C form the data sets from which Fraction of Unbleached TL Signal vs SL Bleach Time curves for each Temperature are plotted later by hand or computer

for TB and PB:

- plateau (Residual and R-dose respectively) plot of ED vs Temperature
- plots (Residual and R-dose respectively) of TL Intensity vs Dose curves for representative temperatures

TL Checklist

	Experiment	Operation	Samples
	Field GC	window data reduced	
		spectral data reduced	
Bulk Sample	Water Content	dried	
		data reduced	
	Lab GC	sample prepared	
		counted	
		data reduced	
	DN	sample prepared	
		run	
		data reduced	
	INAA	sample prepared	
		irradiated	
		gamma counted	
		data reduced	
	XRF	sample prepared	
		run	
		data reduced	
Sample Tube		pretreatment	
		silt separated	
		disks plated	
		IRSL normalized	
	SL	bleached	
		glowed	
		data reduced	
	AF	irradiated	
		preheated	
		glowed, 1st	
		2nd	
		data reduced	
	TB	bleached	
		irradiated	
		preheated	
		glowed	
		data reduced	
	PB	irradiated	
		bleached	
		preheated	
		glowed	
		data reduced	

Pretreatment Work Sheet

Six Samples --->

Date

Tube + Sample (g)
- Tube (205 g)
Total Sample (g)

Tube full?

Stored Sample + Can (g)
- Can (65 g)
Stored Sample (g)

Sample in 600 ml beaker (g)

Cabinet Shelf (top or bottom)

4 N HCl
    H_2O to 500 ml
    (2 hr, aspirate)
    H_2O to 500 ml
    (2 hr, aspirate)

100 ml 30% H_2O_2
    (few hr)
    100 ml 30% H_2O_2
    (overnight, aspirate)
    H_2O to 500 ml
    (2 hr, aspirate)

50 ml 50g/L Na pyrophosphate
    (ultrasound 10 min)
    (overnight)

(H_2O to 300 ml)
    (20 sec, decant thru sieve)
    (H_2O to 300 ml)
    (20 sec, decant thru sieve)
    (H_2O to 300 ml)
    (20 sec, transfer to sieve)

900 ml P230 slurry to 1000 ml beaker
    (store in cabinet)

(wash R230 sand on sieve w/H_2O)
    (R230 sand to 250-ml beaker)
    (several times: H_2O, 20 sec, aspirate)
    (several times: MeOH,20 sec, aspirate)
    (air dry in cabinet)

    R230 Sand + Beaker (g)
    - Beaker (g)
    R230 Sand (g)

    Number of 35-mm film canisters =

Silt Separation Work Sheet

Six Samples --->

Date

(300 ml or 900 ml of 900 ml P230 slurry to 1000 ml graduate)
(H$_2$O to 1000 ml, shake, 60 min)
(decant to 1000-ml beaker)

(30 ml to each of four 40-ml centrifuge tubes)
(7 for 3 sec, 3 for 15 sec)
(aspirate)
(repeat until 100 mg/tube, penny)

Total number of 30 ml aliquots in 4 tubes =

(3 x 15 ml MeOH wash, centrifuge, 7 for 3 sec, 3 for 15 sec, aspirate)

(1 centrifuge tube to 4 oz, 120-ml bottle)
(add MeOH until shadow of finger faintly visible, 1.5-2.0 mg silt/ml)
(check concentration by pipetting 1.00 ml to tared planchet)

Planchet (g)
Planchet + silt (g)
Silt/ml (mg)

(adjust to 100 ml containing 150-200 mg silt /100 ml by adding MeOH and silt)
(prepare second 100 ml bottle using same proportions)

Number of centrifuge tubes =
Volume of slurry =
Number of 100 or 200 ml batches (bottles) =

(settle <11-μm sediment from remaining P230 slurry overnight)
(aspirate)
(transfer <11-μm sediment to 150-ml beaker)
(1 hr, aspirate)
(moist <11-μm sediment to 35-mm film canister)

Canister (7.2 g)
Canister + <11-μm sediment (g)
<11-μm sediment (moist) (g)

TL Disks

Sample: ______________________ Sample Type: sand or loess or other

Filter = ____________________

Experiment (SL,TB, PB,AF)	NDISK	Treatment (N+ m B, h SL, PH) (dates indicate completion)	IRSL Normalize		Glow		
			Normal.File		Run File	Exper.File	
			Date	NRUN	NRUN	Date	NRUN
	1						
	2						
	3						
	4						
	5						
	6						
	7						
	8						
	9						
	10						
	11						
	12						
	13						
	14						
	15						
	16						
	17						
	18						
	19						
	20						
	21						
	22						
	23						
	24						

TL Disks (continued)

Sample: ____________________

Filter = ____________________

Experiment (SL,TB, PB,AF)	NDISK	Treatment (N+ m B, h SL, PH) (dates indicate completion)	IRSL Normalize		Glow		
			Normal.File		Run File	Exper.File	
			Date	NRUN	NRUN	Date	NRUN
	25						
	26						
	27						
	28						
	29						
	30						
	31						
	32						
	33						
	34						
	35						
	36						
	37						
	38						
	39						
	40						
	41						
	42						
	43						
	44						
	45						
	46						
	47						
	48						

SUNLIGHT BLEACHING

Block = ________

Total time desired = ________

Date	Time

Block = ________

Total time desired = ________

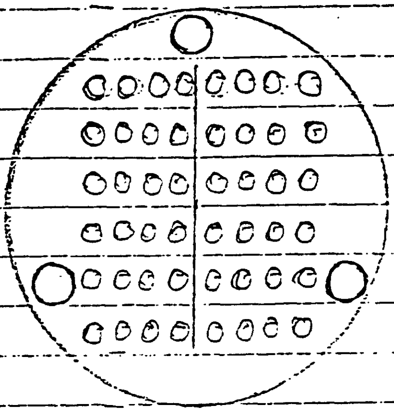
Date	Time

BETA IRRADIATION

NPOS	Sample NDISK's IT (sec)	Sample NDISK's IT (sec)
	Start date,time = ___________	Start date,time = ___________
0- 3		
4- 7		
8-11		
12-15		
16-19		
	Start date,time = ___________	Start date,time = ___________
0- 3		
4- 7		
8-11		
12-15		
16-19		
	Start date,time = ___________	Start date,time = ___________
0- 3		
4- 7		
8-11		
12-15		
16-19		
	Start date,time = ___________	Start date,time = ___________
0- 3		
4- 7		
8-11		
12-15		
16-19		

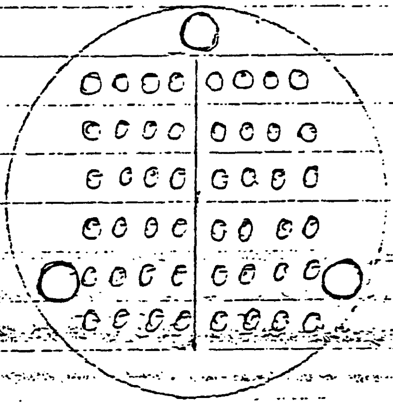
PREHEAT OVEN

Sample	NDISK	Sample	NDISK



Date,Time

Sample	NDISK	Sample	NDISK



Date,Time

Appendix E. Procedures for Separation of Silt from Sample in Sample Tube

E.1. Reagents

deionized (DI) H_2O
4 N HCl
30% H_2O_2
50 g/L Na pyrophosphate dispersant (prepare 2 liters at a time)
methanol

E.2. Apparatus

dark storage cabinet - dark, opaque cloth draped over doors of asbestos-lined oven under hood
ultrasonic bath
beakers
watch glasses
stirring rods
clinical centrifuge (IEC, Model CL)
40-ml plastic centrifuge tubes
Al disks (1-cm diameter x 0.012" thick)
glass shell vials, 0.5 dram, 12x35 mm, flat-bottom (Baxter 87784-1A)
wood vial block
polystyrene cell well boxes with lids, 24 wells, 16-mm well diameter, spray painted black on the outside (Corning 25820)
1.00-ml automatic pipet with tips (Eppendorf)
forceps

E.3. Opening Sample Tube and Removing Sample

Delta-1 Jr. Safelight (red) at least 10-feet away from samples (keep exposure of samples to a minimum)
Note: tare on 35-mm film canister = 7.0-7.4 grams, Ave. = 7.2 grams
Note: tare on 6-inch plastic sample tube + caps + tape = ~205 grams (+-2 grams)
carefully remove caps from ends of tube and record whether tube is full to the ends or not
remove 1 inch of sample (~150 grams) from each end and discard
divide central 4 inches (~300 grams) into 1/3 and 2/3 portions
save 2/3 portion (~200 grams) in 1/2-pint paint can(s) and seal (tare on can + top = 65 grams)
place 1/3 portion (~100 grams, ~100 ml) in 600 ml beaker (tare = 172-180 grams, Ave. = 176 grams)

E.4. Pretreatment (Chemical Treatment and Particle Size Separation)

Delta-1 Jr. Safelight (red) at least 10-feet away from samples (keep exposure of samples to a minimum) if R230 (>63 μm) fraction is to be saved for OSL. Otherwise, Na vapor lights (orange) hanging from ceiling.
add 4 N HCl until entire sample is wet, break up and disperse with stirring rod, and let stand >1 hour in dark storage cabinet to remove carbonate
(50 ml 4N HCl = ~10 grams carbonate)
remove as much supernatant as possible with aspirator (glass tube connected by tubing to trap on tap water suction)

add DI H_2O to 500 ml and let stand >=4 hours in dark storage cabinet (4-μm particles settle at 11 min/cm x 10 cm = 110 min)
remove supernatant with aspirator
repeat washing once more (gives 0.03-0.1 N HCL)

add 20 ml 30% H_2O_2, stir with rod (watch for foaming), and cover with watch glass
if there is no foaming after ten minutes or the foaming has subsided, add additional 20 ml portions of 30% H_2O_2 until a total of 100 ml has been added
allow to sit for few hours in dark storage cabinet
add second 100 ml 30% H_2O_2 and stir with rod (gives ~25% H_2O_2)
let stand overnight in dark storage cabinet to decompose organics
remove supernatant with aspirator
add DI H_2O to 500 ml and let stand >=4 hours in dark storage cabinet
remove supernatant with aspirator (gives ~3% H_2O_2)

add 100 ml Na pyrophosphate (50g/L) as decoagulant (for loess and for sands if silt fraction to be separated for TL)
break up sediment with stirring rod (pH = ~8 ?)
ultrasound for 10 min (sediment settles into very dense mass on bottom of beaker) and store in dark storage cabinet

E.5. Wet Sieving

Delta-1 Jr. Safelight (red) at least 10-feet away from samples (keep exposure of samples to a minimum) if R230 (>63 μm) fraction is to be saved for OSL. Otherwise, Na vapor lights (orange) hanging from ceiling.
place 8-inch 230-mesh stainless steel sieve (63 μm = 0.0025") on plastic bucket (clean sieve between samples by ultrasounding)
add DI H_2O to sample in 600 ml beaker to bring slurry to 300 ml
disperse sediment with stirring rod and stir
allow to settle 20 seconds
(63-μm particles settle at 3 sec/cm x 6 cm = 18 sec)
decant supernatant above settled sediment thru sieve and catch P230 slurry in bucket
add DI H_2O to 300 ml in the 600 ml beaker, disperse sediment again with stirring rod, allow to settle 20 seconds, and pour slurry thru sieve
add DI H_2O to 300 ml in the 600 ml beaker, disperse sediment, and immediately pour thru sieve, transferring sediment to sieve
wash any remaining sediment onto sieve with DI H_2O from wash bottle
wash 600-ml beaker with tap water and rinse with DI H_2O
stir 900 ml P230 (<63 μm) silt + clay slurry in bucket with stirring rod and pour:

sand	loess
300 ml (1/3, ~25-30 grams) back into rinsed 600-ml beaker discard remaining 2/3 of slurry	entire 900 ml into 1000-ml beaker

store P230 (<63 μm) silt + clay slurry) in covered 600-ml or 1000-ml beaker in dark storage cabinet

if R230 (>63 μm) sediment on sieve is to be saved for OSL, wash with tap water to remove any remaining P230 particles (don't save, allow to go down drain) and wash to one side of sieve
transfer R230 (>63 μm) sand fraction to 250-ml beaker with methanol
wash several times with methanol and decant with aspirator
allow methanol to evaporate for several days in dark storage cabinet
transfer R230 (>63 μm) sand fraction to 35-mm film canisters

E.6. Separation of 4-11 μm Silt for TL

use Na vapor lights (orange) hanging from ceiling

Stoke's settling:
transfer P230 (<63 μm) suspension to 1000 ml plastic graduated cylinder
add DI H_2O to 1000 ml
cover with Parafilm and shake (or stir with long stirring rod), and let stand 50 min (at 23°C)
(11-μm particles settle at 90 sec/cm x 36 cm = 54 min)
decant suspension (<11 μm) into 1000-ml beaker

Note: could repeat settling with sediment (11-63 μm) once more to increase yield but then need to settle overnight to concentrate slurry)
store suspension (<11 μm) in 1000-ml beaker in dark storage cabinet
discard sediment (11-63 μm)

Note: if the clay content is very high, the subsequent separation of the 4-11 μm silt fraction by centrifugation will be perturbed. Get rid of the clay in advance by letting the suspension settle in the 1000-ml beaker for >2.5 hours, aspirate and discard the supernatant. Repeat as often as necessary until the supernatant is clear.

Centrifugation of silt:
pour 30 ml of suspension (<11 μm) into each of four 40-ml centrifuge tubes and centrifuge (flip setting to 7 for 3 sec and then lower setting to 3 for 15 sec (1390 rpm))
remove supernatant (<4 μm, clay) with aspirator and discard
repeat until there are ~500 mg of sediment in each tube (circle in bottom of tube about size of penney)
wash sediment (4-11 μm, silt) three times with 30 ml DI H_2O, centrifuge on 7 after each wash, and remove supernatant with aspirator
then wash sediment (4-11 μm, silt) three times with 15 ml reagent grade methanol, centrifuge on 7 after each wash, and remove supernatant with aspirator
transfer sediment from a centrifuge tube to an 8 oz. (238 ml) bottle with methanol
add reagent grade methanol until shadow of finger held behind bottle is faintly visible when bottle is held up to Na vapor light (1.5-2.0 mg of sediment/ml)
check concentration of silt gravimetrically by pipeting 1.00 ml with Eppendorf pipet onto tared beta counting planchet, evaporate methanol in 40°C oven, and weigh to determine mg of sediment/ml of slurry
repeat with other centrifuge tubes until ~200 ml of suspension in bottle
fine silt (4-11 μm) suspension ready to plate on disks

allow remaining suspension (<11 μm) to settle overnight in dark storage cabinet
(3-μm particles settle at 17 min/cm x 9.5 cm = 162 min)
remove supernatant (clay) with aspirator
transfer sediment to 150-ml beaker with DI H_2O and allow to settle for an hour
remove supernatant (clay) with aspirator
transfer moist sediment to tared 35-mm film canister
weigh and record mass of fine silt (<11 μm, most clay removed) + remaining H_2O (ignore)
seal film canister with black tape and store moist sediment (<11 μm) in case additional fine silt (4-11 μm) must be prepared for plating on disks

Appendix F. Procedures for Preparing and Normalizing Disks for TL

F.1. Preparation of Disks

1-cm diameter Al disks punched from 0.012"-thick 1100 aluminum sheet using Di-Acro #1 single station punch, lubricated with WD-40
remove any burrs on edges with sand paper
remove oil and fingerprints with MIBK and ultrasound
decant MIBK and wash and dry with acetone

F.2. Plating Disks

have 100 ml of 4-11 μm suspension in sufficient methanol in one or two 4 oz (120 ml) glass bottles to give 1.7-2 mg/ml (can faintly see shadow of finger thru suspension in bottle when bottle held up to Na vapor light)
place 72 or 96 flat-bottomed shell vials in vial blocks and put a 1-cm diameter Al disk in the bottom of each tube with forceps (don't handle disks with fingers)
Note: can add Teflon-coated stirring magnet to 4-oz bottle and place on magnetic stirrer to keep sediment in suspension or simply shake bottle after each pipeting. Drag magnet from bottle after use with another magnet.
adjust concentration of suspended sediment in bottle to 1.5-2 mg/ml
pipet 1.00 ml of the suspended sediment (1.5-2 mg) into each tube with the Eppendorf pipet
allow the methanol to evaporate in 40°C oven overnight
remove the plated Al disks by turning shell vial upside down and gently tapping disk out onto a paper towel
pick up disks with forceps and transfer to plastic cell well box (24 disks per box, boxes spray painted with black paint, bottom edges sealed with black tape to exclude light, and white label on top)

seal top of 4 oz bottles, containing remaining sediment slurry, with black tape to prevent evaporation
mark level of slurry with Sharpie in case methanol evaporates
store bottle in bottom rear of storage cabinet in case more disks need to be prepared in near future

F.3. Normalization of Disks by IRSL

The disks are normalized for intercomparison by a 5-second exposure to infra-red illumination and detection of the resulting luminescence by a photomultiplier covered with Schott BG-39 and Kopp 7-59 filters (as well as the Pyrex window).

F.4. Normalization of Disks by Reirradiation and Glowing

The disks are normalized for intercomparison by reirradiating for 5 minutes in the beta irradiator and reglowing using Schott UG-11 and Corning CS1-75 filters or other appropriate filters.

Appendix G. Procedures for Operating Beta Irradiator

Manuals:
Daybreak Model 801 Multiple Sample Irradiator Manual, Revision 2 (September 18, 1985)

To turn on:
turn key (right front), gives:
Daybreak 800/801
ENTER, LOAD, RUN, CE? (Main Menu)
carousel moves until position #10 is under Load Door,
#00 (#10 + 10) under beta source, and
#02 (#10 + 12) under alpha source

To terminate any mode and return to Main Menu:
press EXIT

To load sample disks (up to 20 disks):
press LOAD and remove Load Door
carousel rotates so position #00 under Load Door
carousel positions are numbered clockwise
press arrows to move Carousel one position:
> = advance one sample #
< = backup one sample #
load sample disks with forceps onto inserts in the carousel (samples must be <1.0 cm x <0.025 inches thick)
press EXIT to return to Main Menu

To operate in Multiple Mode:
set MULTIPLE/SINGLE switch to MULTIPLE and INTERNAL/EXTERNAL switch to INTERNAL

To enter data in Multiple Mode:
press ENT
press A/B to toggle between A (alpha) and B (beta) irradiation for each sample #
enter TS (time set, units of 10 seconds, <= 9999 seconds or ~28 hours) into display (seconds) for each sample # (#00 to #19) - enter 0 seconds if this sample # not to be irradiated
press ENT to enter number or CE (cancel entry) to cancel
sample # increments after TS entered
press arrows to change sample #:
> = advance one sample #
< = backup one sample #
press EXIT to return to Main Menu, entry table prints out sample #, TS (time set, seconds), and A or B

To clear all data:
press CE (clear entries)
press: EXIT (complete clearing)
or, > (escape without clearing)

To run irradiation cycle in Multiple Mode:
press RUN, carousel positions first sample disk with non-zero TS entry under shutter, shutter clicks open, exposure starts, and remaining exposure time (units of 10 seconds) counts down
press STOP to stop irradiation and RUN to resume irradiation of this sample or EXIT to advance to next sample

after irradiation, printer prints sample # irradiated, TS (time set, units of 10 seconds), and ET (elapsed time, units of 10 seconds)
carousel advances to next sample # with non-zero TS entry
after completion of the cycle, the data table is cleared and the carousel moves to position #00

errors: SHUTTER NOT OPEN - press RUN to try again or EXIT to return to Main Menu (remove side panels to inspect irradiators)
SHUTTER STUCK OPEN - power down instrument and get help before opening instrument

To operate in Single Mode:
set MULTIPLE/SINGLE switch to SINGLE and INTERNAL/EXTERNAL switch to INTERNAL

To enter data in Single Mode:
press ENT
enter sample #
press A/B to toggle between A (alpha) and B (beta) irradiation
enter TS (time set, units of 10 seconds, <= 9999 seconds or ~28 hours) into display (seconds)
press ENT to enter number, entry prints out sample #, TS (time set, seconds), A or B, and returns to Main Menu

To run Single Mode irradiation:
press RUN, enter sample #
carousel positions sample disk under shutter, shutter clicks open, exposure starts, and remaining exposure time (units of 10 seconds) counts down
press STOP to stop irradiation and RUN to resume irradiation of this sample or EXIT to return to Main Menu
after irradiation, printer prints sample # irradiated, TS (time set, units of 10 seconds), and ET (elapsed time, units of 10 seconds)
after completion of the irradiation, the data table is cleared and the carousel moves to position #00

errors: SHUTTER NOT OPEN - press RUN to try again or EXIT to return to Main Menu (remove side panels to inspect irradiators)
SHUTTER STUCK OPEN - power down instrument and get help before opening instrument

Appendix H. Procedures for TL Experiments

H.1. Preheat

used in test for anomalous fading and total bleach and partial bleach TL methods

disks heated at 124°C for 64 hours on aluminum plate inside calibrated hotplate oven

H.2. Determination of Sunlight Bleach Sensitivity (36 Disks)

expose sets of 4 natural disks to full sunlight for each of 9 bleach times

N
N, 5 m SL (natural dose, 5 minute bleach in sunlight)
N, 10 m SL
N, 30 m SL
N, 1 h SL
N, 2 h SL
N, 6 h SL
N, 16 h SL (assumed to be residual level)
N, 32 h SL

glow disks
plot normalized TL Signal vs Bleach Time curve

H.3. Test for Anomalous Fading (16 Disks)

irradiate each set of 4 disks for 1 hour
(Note: time irradiations, preheats, and storage so all 16 disks are glowed at the same time.)

sequence:

1) preheat 4 disks for 64 hours at 124°C, store for 4 weeks, and glow
   (N + 1h B, PH, Stored)
2) no preheat, store 4 disks for 4 weeks, and glow
   (N + 1h B, no PH, Stored)
3) preheat 4 disks for 64 hours at 124°C and glow immediately
   (N + 1h B, PH, Immed)
4) no preheat, glow immediately
   (N + 1h B, no PH, Immed)

compare shapes of TL glowcurves using Daybreak Anomalous Fading program in TL Compute menu

(no PH,Stored) / (no PH,Immed) and (PH,Stored) / (PH,Immed)
gives rate of anomalous fading and tells whether preheat removes component responsible for anomalous fading

(PH,Immed) / (no PH,Immed) and (PH,Stored) / (no PH,Stored)
should agree (tells whether PH too intense)

H.4. Total Bleach Method (24 Disks)

set aside 4 natural disks (N)
bleach 4 natural disks in sunlight for time required to reach residual level (N, 16 h SL)
irradiate 4 disks for each of 4 dose data points in beta irradiator (~300 rad/min, ~3 Gy/min):

N + (EDT)m B (natural dose + EDT minutes beta, where EDT = minutes in irradiator which gives dose equivalent to ED)
N + (2 x EDT)m B
N + (4 x EDT)m B
N + (8 x EDT)m B

preheat irradiated disks (as one set)
store for 1 week
glow disks
use Daybreak program in TL Compute menu to plot unbleached TL Signal vs Dose data and to fit best curve
extrapolate unbleached TL Signal vs Dose curve to intersection with residual level (N,16 h SL) to get total bleach ED

H.5. Partial Bleach Method (20 Disks)

set aside 4 natural disks
irradiate 4 disks for each of 4 dose data point in beta irradiator (~300 rad/min, ~3 Gy/min):

N + (EDT)m B (natural dose + EDT minutes beta, where EDT = minutes in irradiator which gives dose equivalent to ED)
N + (2 x EDT)m B
N + (4 x EDT)m B
N + (8 x EDT)m B

bleach all disks in sunlight for the time required to reduce natural TL signal halfway to residual level (e.g., N, 1h SL and N + 1h B, 1h SL)
preheat irradiated disks (as one set)
store for 1 week
glow disks
use Daybreak program in TL Compute menu to plot TL Signal vs Dose curve and to fit best curve
intersection of unbleached TL Signal vs Dose curve from total bleach method with bleached TL Signal vs Dose curve from partial bleach method gives partial bleach ED

Appendix I. Procedures for Daybreak TL Instrument and TLAPPLIC Software

I.1. General Information

Manuals:
Daybreak Sytem TLAPPLIC TL Applications Software Manual (Version 3.23)
TLAPPLIC Version 3.2 for Daybreak Model 1100 Automated TL System
1100 Installation Manual (Version 0.0, 21 May 1990)

File organization (NRM refers to normalization):
samplename.PRG files (and samplenameNRM.PRG) = batch program
(GEN.PRG and GENNRM.PRG = generic programs)
samplename.DAT files (and samplenameNRM.DAT) = raw data
samplename.SUB files (and samplenameNRM.SUB) = subtracted data
record = 1 glowcurve
data file <= 80 glowcurves (1 sample, at least 1 glowcurve must be a background)
data directory <= 32 data files (set of samples)

To start TLAPPLIC software from COLOR mode:

```
> TL                              (switches to TL mode)
system will request CR,CR to perform reboot here
enter 14, 4,CR, N, N, Y   (for Grafplus)
gives "Welcome ..."
turn printer ON if not on already
hit key or click left mouse button
gives Main TLAPPLIC Menu
```

To start TLAPPLIC software if already in TL mode:

```
> cd \DAYBREAK
> TLAPPLIC
```

To exit TLAPPLIC program:
select "Exit (to DOS)" from Main TLAPPLIC Menu

To select functions from menus and make entries:
type first letter of word (no CR)
or, click left mouse button on first letter of word

Note: mouse cursor is constrained to first letters of words.

space = Y (usually)
CR required after numeric entry

mouse cursor = block with inverted character
text cursor = blinking underline
graphics cursor = arrow

Definitions of various identification parameters:

batch = one carousel loading of disks from a sample
NRUN = run number (record number for glowcurve in data file)
BRUN = batch run number for glowcurve (matches record number in batch program, first record always = 0)
NDISK = disk number assigned during preparation of disks (e.g., 1 to 96 for each geological sample), these must be in sequence for a batch program

NDISK0 = starting disk number corresponding to first position in carousel (POS#=0), assigned when data file is opened (will be fictitious if POS#0 > 0)
POS# = position in carousel (= 0-19)
POS#0 = position in carousel corresponding to first record in batch program (BRUN=0), assigned when creating or editing batch program

Note: NDISK = NDISK0 + POS#

Note: can change POS# in middle of run by selecting "Advance dsk" or "First dsk" (POS# and NDISK change but not BRUN#)

Strategy for setting up TL batch runs:

1) after disks are plated, they are placed in four plastic cell well boxes (24 disks disks per box) and assigned unique sequential numbers (NDISK = 1-96)

2) groups of disks (4 disks per data point) are carried thru various TL experiments and methods:

	data points	number of disks	
SL - sunlight bleach sensitivity	9	36	
AF - test for anomalous fading	4	16	
TB - total bleach TL method	6	24	
PB - partial bleach TL method	5	20	(plus 5 data points for 20 disks from total bleach = 40 disks)
	Total	96	

3) only 10 data points or 40 disks can be treated in a single .DAT file (40 primary glowcurves + 40 backgrounds = 80 records)

4) the disk numbers (NDISK) must be sequential in any batch run. Thus, unless care is taken to choose contiguous disks for any given experiment, several batch runs may exist for an experiment (filenameA.DAT, filenameB.DAT, etc.)

5) before any treatment or data reduction is performed on the batch run .DAT files, these files must be reorganized into appropriate experiment .DAT files using MERGE (merged files still limited to 40 disks per file)

Example of organization of sample disks into batches:

1st Batch - preliminary run on natural TL and natural + beta irradiation TL disks (starting disk number = 0)

NRUN	BRUN	NDISK	POS#	Treatment	
0	0	1	0	N	(natural TL)
1	1	1	0	Bkg	
2	2	2	1	N	
3	3	2	1	Bkg	
4	4	3	2	N	
5	5	3	2	Bkg	
6	6	4	3	N	
7	7	4	3	Bkg	
8	8	5	4	N + 15m B	(15 m Beta irrad)
9	9	5	4	Bkg	
...					
14	14	8	7	N + 15m B	
15	15	8	7	Bkg	
16	16	9	8	N + 30m B	(30 m Beta irrad)
17	17	9	8	Bkg	
...					
22	22	12	11	N + 30m B	
23	23	12	11	Bkg	
24	24	13	12	N + 60m B	(60 m Beta irrad)
25	25	13	12	Bkg	
...					
30	30	16	15	N + 60m B	
31	31	16	15	Bkg	

2nd Batch - sunlight bleached disks and repeat from 1st Batch (starting disk number = 17)

NRUN	BRUN	NDISK	POS#	Treatment	
32	0	17	0	N + 16h SL	(16 h sunlight)
33	1	17	0	Bkg	
...					
38	6	20	3	N + 16h SL	
39	7	20	3	Bkg	
40	8	21	4	N + 30m B	(30 m Beta irrad)
41	9	21	4	Bkg	
...					
46	14	24	7	N + 30m B	
47	15	24	7	Bkg	

I.2. Data Collection

To change operating parameters (customizes INFO file):

Note: when TLAPPLIC software is started, operating parameters are taken from the INFO file (these are altered when an existing .DAT file is opened)

select "Parameter update" from Main TLAPPLIC Menu
gives Change Parameters menu (see figure 33)
select items and enter or toggle in new values (see Table 18):
select "Other parameters for the 1100 TL system",
gives Automated TL Parameters menus (see figure 34)
select items and enter or toggle in new values (see Table 19)

Table 18. Commonly used values for "Parameters" .

Alpha:	0.0000	u-2/sec Am-241	on 27-AUG-92	halflife=	432.0 (yr)
Beta:	2.79	Gy/min Sr-90	on 01-JAN-93	halflife=	28.8 (yr)
Gamma:	0.0000	Gy/sec Co-60	on	halflife=	5.26(yr)
Irrad date:					
Mode of ramp:	REPEAT				
Ramprate: (C/sec)	5.0				
Scale:	10000.0				
Default DIRECTORY name:	tldata\				
Limits for integrals: (C)	200 to 500				
Will BLEACH samples:	NO				
Number of records to reserve in opening new file:	80				
Correct for source decay:	YES				
Figure in DEADTIME correction:	YES	50 nsec			
Has IBM charset for printer:	YES				
Port I/O address for interface:	912				
Type of curve filter:	3 pt gaussian				
Preheat: (C)	145 for 12 sec				

Table 19. Commonly used values for "Other parameters".

Irradiator type:	external	internal, no elevator
Top BETA doserate =		~0.022 Gy/sec
Low BETA doserate =		0.0
HighBETA doserate =		0.0
What BETA:		internal w/o elevator
Mode of irrad:		batch irrad-signal operator
Normalize after batch:	NO	
Xposure time for norm: (sec)	0	
Preheat (norm) at 0C for	0	
Analysis type:		standard growth curve
Cool temperature (C)		60
Ramp max temp (C)	300	500

Before taking data, need to:
1) plan run (including backgrounds and any normalizations) and have TL Disks sheets available
2) load sample disks onto carousel (batches of up to 20)
3) make sure correct filters are in front of photomultiplier tube (see Table 20 and figure 6)
4) generate a batch program
5) open a data file

Table 20. Filters for the TL apparatus. Suppliers of the filters are Schott (BG-39 and UG-11), Kopp (7-59), and Corning (CS1-75). Early Work included samples MMV-5, 6, 7, and 9, while Middle Work included samples NVP-2, NEC-17, 18, 19, and 20, and MCS-3a and 3b. All other samples were run under the Current Setup for IRSL and TL.

	Early Work	Middle Work	Current Setup for IRSL and TL
Spacer	spacer	spacer	spacer
Upper	UG-11	BG-39	BG-39
Lower	CS1-75	7-59	7-59 IRSL adapter Pyrex window

To open TAKE1100 command menu:
select "Take data: 1100 auto system",
gives Change Parameters menu (see figure 33)

To generate a batch program:
batch program consists of sequence of records which provide information about each disk and define treatment of disk in each carousel position
each batch program record leads to a glowcurve record in the data file
select "Program select/generate" in TAKE1100 command menu,
gives Program 1100 menu and submenus (see figures 35, 36, 37, 38, and 39)
enter data for each sample disk

To open a data file:
select "Open data file" in TAKE1100 command menu,
gives Change parameters menu

To begin physical operation of system:
start vacuum pump
open main valve on nitrogen tank

select "Go/resume" from TAKE1100 command menu

select "Vacuum" -
bleed valve opens
main vacuum valve opens when ~1 mmHg (upper left of screen)
when <50 uHg, select "Vacuum" again to close vacuum valve

select "Purge" (immediately) -
purge valve opens (flowmeter = 4 cu.ft./hr = 2 L/min)

select "HV" -
turns on HV to PMT (dark count rate = 40-80 cps)

To clean oven (start of runs for day), run a ramp:
click right button on mouse (starts TLCONSOLE, see TLAPPLIC-13)
select "Txt_entry:"
enter G1, CR - starts ramp
enter G0, CR - stops ramp (don't go above 500°C)
CR (exits Txt_entry)
select "X" (exit TLCONSOLE)

To begin acquiring data:
select "Go/continue" from TAKEDATA screen (see figure 40),
gives version of TAKEDATA screen with Data Acq commands (see figure 41)

Note: background is automatically subtracted (using background for previous disk) and subtracted glowcurve written to .SUB file.

start TLCONSOLE (right mouse button):
select "H" to turn off HV
close Main Valve on nitrogen tank and allow pressure to bleed down to zero on both pressure gauges on regulator
select "P" to close purge valve
select "X" (exit)

select "Exit TAKE1100" in TAKE1100 command menu

To normalize disks using IRSL (before glowing):
make sure IRSL adapter is plugged in
load sample disks onto carousel (batches of up to 20)

select "OSL_takedata:1100 Auto System" from TAKE1100 command menu

select "Change Parameters"
"Repeat" for Ramp Mode is ignored in IRSL mode
ROI = 0-5 channels (for later integration)
OSL Parameter menu:
0 10 msec
0 100 msec
5 1 sec
0 10 sec
LED Current = 20 ma
Sample temperature = 30°C
select "Program Select/Generate"
IRSLNRM1.PRG (20 samples) and
IRSLNRM2.PRG (16 samples) are generic .PRG files

select "Open Data File"
samplenameNRM1.DAT (60 samples) and
samplenameNRM2.DAT (36 samples) will hold data for 96 samples

select "Measure BG" (turns on HV automatically)
select "Start BG" (accumulates background, should be 60-120 counts/sec)
select "OK"

Note: very important, gives BG countrate before each batch of 20 disks

select "Go/Resume"
only HV is ON (from background measurement)

select "Go/Continue"

To normalize disks by reirradiating (after glowing):
using external beta irradiator - reirradiate in Daybreak 801 Irradiator for 5 min

select "Normalize program generate" in TAKE1100 Command Menu

select "Change parameters" if necessary,
gives 2ND GLOW NORMALIZATION MENU
enter parameters (see Table 21) for:
don't irradiate for normalization in TL instrument (external beta irradiator)
No
No
File ID
Max Disk #
5
External
0 0
500°C

select "Continue"

"If params at right are OK and you wish to install NORM program in memory, hit INSTALL"

open data file (samplenameNRM.DAT) same as for sample disks

take glowcurves in same way as for sample disks

Table 21. Commonly used values for normalization parameters.

	don't irradiate for normalization in TL instrument	irradiate for normalization in TL instrument	
		manually	automatically
Do normalization:	NO	YES	YES
Automatic:		NO	YES
File id:			
Max dsk# (0-19):	(actually maximum position number)		
Irrad time: (min)		30	30
Type of beta irrad:	EXTERNAL	TOP	TOP
Preheat at 250C for (sec)		0	0
Ramp max temp: (C)		450	450

I.3. Data Reduction

To merge or reorganize data files:

Note: used to convert:
batch run .DAT files to experiment .DAT files, and
batch run NRM.DAT files to experiment NRM.DAT files.

Note: can't exceed 80 records in destination file and this file can't exist before merge.

select "Merge and/or edit files" in Main TLAPPLIC Menu, gives TL file management menu:

select "Merge files", enter destination filename

click on source filename.DAT in directory, gives TLMERGE menu

select "Select curves to copy" and click on NRUN numbers (include background record for each glowcurve)

select "Copy curves to dest" (glowcurve and background records transfer to destination .DAT file and subtracted records transfer to destination .SUB file)

select "New source file" to move on to another source file

select "Exit" to finalize transfers

To print .DAT file:
select "Compute" in Main TLAPPLIC Menu

click on filename.DAT in directory
select "Continue"

select "Continue",
gives File synopsis: Continue
Print
select "Print"

To compute data from TL glowcurves:
select "Compute" in Main TLAPPLIC Menu

select "new Filename" or click on filename.DAT in directory or use current filename.DAT
select: "Continue",
gives File synopsis: Continue
Print

gives Compute menu:

"Look" = display glowcurves

"Subtract" = shift glowcurves up or down in temperature and subtract background

"Plateau" = plot graph of equivalent dose for a range of temperatures with standard deviations

"Check Fade" = compare two sets of identical sample disks to evaluate anomalous fading (one set glowed immediately after irradiation, the other set glowed after decay time), or compute ratio between them

"Normalize" = enter New NORM factor for each NRUN

"Growthcurve" = find ED for single temperature using saturating exponential or linear fit

Note: natural TL glowcurves (zero dose) given double weight during fits.

"Integrals" = gives PRINT DATA menu:
- Raw data
- Subtracted data
- Integrate all curves
- Normalization data
- Page printer
- Display file synopsis
- Exit

"File..open new" = gives COMPUTE: NEW FILE menu:
- new Directory name
- new File name
- Continue
- Exit

"Exit"

Overview of initial treatment of all glowcurves:

```
      "Compute"
      "Print"                      - print synopsis of .DAT file
      "Subtract", "Subtract"       - subtract backgrounds from each glowcurve
      "Look", "Raw", "Print"       - print out several representative raw + bkg
                                          glowcurve plots
      "Look", "Subtracted"         - select a group of glowcurves
                        "Print"    - print Bkg Subtracted glowcurve plots
      "Normalize"                  - enter normalization factors
      "Look", "Subtracted"         - select a group of glowcurves
                        "Print"    - print Bkg subtracted and Normalized
                                          glowcurves
      "Look", "Subtracted"         - select a group of glowcurves
                        "Tempshift" - T shift glowcurves until they line up
                                          (corrects for differences in thermal
                                          inertia among disks)
                        "Print"    - print Bkg Subtracted, Normalized, and
                                          T Shifted glowcurve plots
```

To compute normalization factors (relative to average integral value) from IRSL shinedown curves:

```
      select "Compute" in Main TLAPPLIC menu
      enter samplenameNRM1.DAT or samplenameNRM2.DAT


      "Look", "Raw", "Print"       - print out several representative
                                          raw + bkg shinedown curves
      "Look", "Subtracted"         - select all shinedown curves
                        "Print"    - print Bkg subtracted shinedown curves
      "Integrals/Utilities", "Integrate All Curves by ROIs"
                                   - gives printout of integrals for each NRUN
      "Exit"

      average integrals by hand
      compute normalization factor for each NRUN
```

To compute normalization factors (relative to a single glowcurve) from TL normalization glowcurves:

```
      choose temperature range for integration from glowcurve plots
      "Integrals", "Normalization data"
            enter T limits for integration
            enter NRUN to be used for normalization
            gives printout of normalization factors for each NRUN
      "Exit"
```

To compute normalization factors (relative to an average integral value) from TL normalization glowcurves:

```
      choose temperature range for integration from glowcurve plots
      "Integrals", "Integrate all curves"
            enter T limits for integration
            select "Subtracted",
                  gives printout of integrals for each NRUN
      "Exit"
            average integrals by hand
            compute normalization factor for each NRUN
```

To apply normalization factors to primary glowcurves:
"Normalize" - enter normalization factor for each NRUN
"Look", "Subtracted" - select a group of glowcurves
"Print" - print Bkg Subtracted, T shifted, and Normalized glowcurve plots

To treat sunlight bleaching experiment:
"Check Fade" - treat disk sets for each bleachtime separately
select NRUN's and RUNTYPE's for
N,bleachtime SUN ("Aged") and N ("Fresh")
print out Ratio vs. T plots and data
underline ratios for 200°, 250°, 300°, 350°, 400°C

plot values for each T on bleachtime vs. ratio graph (sunlight bleaching curves for each T)

To treat anomalous fading experiment:
"Check Fade" - used because it generates ratios between sets of disks vs. T
select NRUN's and RUNTYPE's for:
1) Aged and Fresh (anomalous fading)
or, 2) illuminated ("Aged") and unilluminated ("Fresh")
print out Ratio vs. T plot and data

To treat total bleach TL experiment:
"Growthcurve"
select NRUN's to discard
gives GROWTHCURVE ANALYSES POSSIBLE table (indicates whether data needed for various types of growthcurves is present)
click,
gives GROWTH CURVE ANALYSIS OF: filename.DAT (allowed computations are highlighted)
select "RESIDUAL" computation,
gives BLEACH CHOICE
select bleachtime (defines set of NRUN's for residual level),
gives CHANGE TYPE OF FIT menu
select "SATURATING EXPONENTIAL FIT",
gives SATURATING EXPONENTIAL FIT menu
select "ALL TEMPERATURES",
gives ED vs. T curve
select "Print all"
select "SINGLE TEMPERATURE"
enter T,
gives Intensity vs. Dose growth curve for T and Residual Intensity (points can be deleted)
select "Print all"

To treat partial bleach TL experiment:
"Growthcurve"
select NRUN's to discard
gives GROWTHCURVE ANALYSES POSSIBLE table (indicates whether data needed for various types of growthcurves is present)
click,
gives GROWTH CURVE ANALYSIS OF: filename.DAT (allowed computations are highlighted)
select "R-DOSE" computation
... (same as total bleach TL method)

Appendix J. Computing Concentrations of K, U, and Th from Counting Data Collected with the Portable Gamma Spectrometer

J.1. Spectrum Stripping Method

(see Lovborg and Mose, 1987, p. 556; Killeen, 1979, p. 176)

Definitions:

nK,nU,nTh - counts in each window
CT - counting time
bnK,bnU,bnTh - background counts in each window (due to cosmic rays, Rn in air, NaI crystal)
bCT - background counting time
cpsK,cpsU,cpsTh - cps in each window
bcpsK,bcpsU,bcpsTh - background cps in each window

Count rate for each window:

cpsK = nK/CT   bcpsK = bnK/bCT
cpsU = nU/CT   bcpsU = bnU/bCT
cpsTh = nTh/CT   bcpsTh= bnTh/bCT

Background corrected count rate for each window:

bcorrcpsK = cpsK - bcpsK
bcorrcpsU = cpsU - bcpsU
bcorrcpsTh = cpsTh - bcpsTh

Equations identifying sources of counts in each window:

cpsK = bcpsK + cpsKK + cpsUK + cpsThK
cpsU = bcpsU + cpsKU + cpsUU + cpsThU
cpsTh = bcpsTh + cpsKTh + cpsUTh + cpsThTh

(where e.g., cpsUK is the count rate for U in the K window)

Ordinarily, the contribution of K to the U window (cpsKU) and the contributions of K and U to the Th window (cpsKTh and cpsUTh) are either very small or zero and will be ignored. Therefore,

cpsK = bcpsK + cpsKK + cpsUK + cpsThK
cpsU = bcpsU + cpsUU + cpsThU
cpsTh = bcpsTh + cpsThTh

Solving for the count rates for the primary element in each window:

cpsKK = (cpsK-bcpsK) - cpsUK - cpsThK
= bcorrcpsK - cpsUK - cpsThK

cpsUU = (cpsU-bcpsU) - cpsThU
= bcorrcpsU - cpsThU

cpsThTh = (cpsTh-bcpsTh)
= bcorrcpsTh

(These are net count rates for the primary element in each window after subtracting the window backgrounds and interferences due to other elements in the window.)

If:

```
     gamma  =  U counts in K window / net U counts in U window
            =  cpsUK / cpsUU

     beta   =  Th counts in K window / net Th counts in Th window
            =  cpsThK / cpsThTh

     alpha  =  Th counts in U window / net Th counts in Th window
            =  cpsThU / cpsThTh
```

Then (reversing the order and substituting):

```
  cpsThTh =                                 bcorrcpsTh

  cpsUU    =                bcorrcpsU   -  (alpha)(cpsThTh)
           =                bcorrcpsU   -  (alpha)(bcorrcpsTh)

  cpsKK =
      bcorrcpsK - (gamma)(cpsUU) - (beta)(cpsThTh)
    = bcorrcpsK - (gamma)[bcorrcpsU-(alpha)(bcorrcpsTh)] - (beta)(bcorrcpsTh)
    = bcorrcpsK - (gamma)(bcorrcpsU) - [beta-(alpha)(gamma)](bcorrcpsTh)
```

The following efficiency constants are needed to convert net cps to concentration:

```
     efficK     =  1/sensitivity K     =  percentK / cpsKK
     efficU     =  1/sensitivity U     =  eppmU / cpsUU
     efficTh    =  1/sensitivity Th    =  eppmTh / cpsThTh
```

Thus:

```
  percentK = (efficK)(cpsKK)

    = (efficK) { bcorrcpsK    - (gamma)(bcorrcpsU)
                            - [beta-(alpha)(gamma)](bcorrcpsTh) }

    = (efficK) { (cpsK-bcpsK) - (gamma)(cpsU-bcpsU)
                            - [beta-(alpha)(gamma)](cpsTh-bcpsTh) }

    = (efficK)(cpsK) - [(efficK)(gamma)](cpsU)
                            - [(efficK)(beta-(alpha)(gamma))](cpsTh)
      - (efficK) { bcpsK + (gamma)(bcpsU) + [beta-(alpha)(gamma)](bcpsTh) }

  eppmU    =  (efficU)(cpsUU)

    = (efficU) {                (bcorrcpsU)   -  (alpha)(bcorrcpsTh) }

    = (efficU) {               (cpsU-bcpsU)   -  (alpha)(cpsTh-bcpsTh) }

    =                        (efficU)(cpsU) - [(efficU)(alpha)](cpsTh)
                           - (efficU) { bcpsU + (alpha)(bcpsTh) }

  eppmTh   = (efficTh)(cpsThTh)

    = (efficTh) {                                  (bcorrcpsTh) }

    = (efficTh) {                                  (cpsTh-bcpsTh) }

    =                                 (efficTh)(cpsTh)
                                    - (efficTh)(bcpsTh)
```

J.2. Simultaneous Equation Method

(see e.g., Duval, 1989)

Definitions: same as stripping method
bpercentK,bppmU,bppmTh - backgrounds for each element expressed in concentration units

Simultaneous equations:

$$\mathrm{percentK} = -\,\mathrm{bpercentK} + (\mathrm{akk})(\mathrm{cpsK}) - (\mathrm{aUK})(\mathrm{cpsU}) - (\mathrm{aThK})(\mathrm{cpsTh})$$

$$\mathrm{eppmU} = -\,\mathrm{bppmU} + (\mathrm{aUU})(\mathrm{cpsU}) - (\mathrm{aThU})(\mathrm{cpsTh})$$

$$\mathrm{eppmTh} = -\,\mathrm{bppmTh} + (\mathrm{aThTh})(\mathrm{cpsTh})$$

(where aKK, aUK, aThK, etc. are coefficients for the terms)

Note: the values found for the background terms (bpercentK, bppmU, and bppmTh) from the calibrations done on the DOE pads in Grand Junction are quite large because they are residuals from fitting the large concentrations of the elements. They are inappropriate for counting done in field situations where low concentrations of these elements prevail. Therefore, counter backgrounds are subtracted from the cps for each window and then the background terms are ignored.

J.3. Correspondence Between the Coefficients of Terms for the Spectrum Stripping Method and the Coefficients for the Simultaneous Equation Method

By inspection:
aKK = efficK
aUK = (efficK)(gamma)
aThK = (efficK)[beta-(alpha)(gamma)]
bpercentK= (efficK) { bcpsK + (gamma)(bcpsU) + [beta-(alpha)(gamma)](bcpsTh)}

aUU = efficU
aThU = (efficU)(alpha)
bppmU = (efficU) { bcpsU + (alpha)(bcpsTh) }

aThTh = efficTh
bppmTh = (efficTh)(bcpsTh)

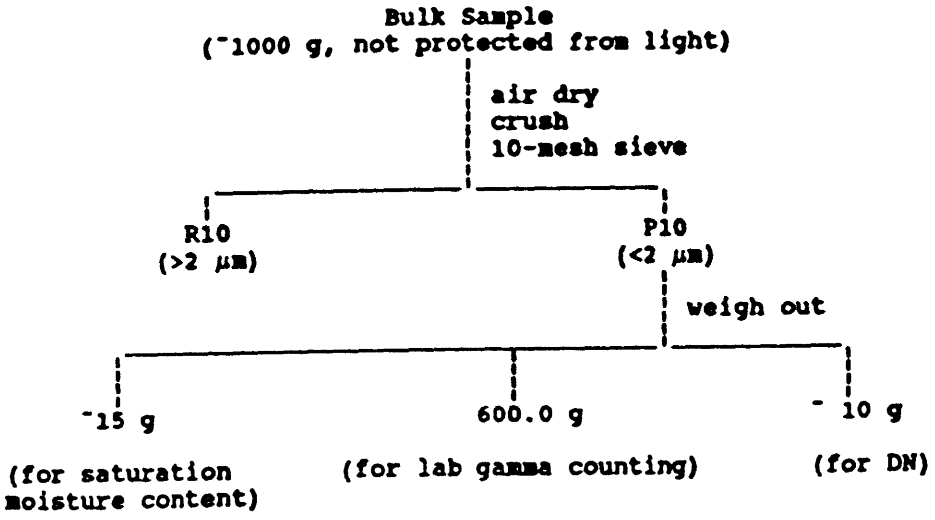


Figure 1. Flow diagram for the bulk sample.

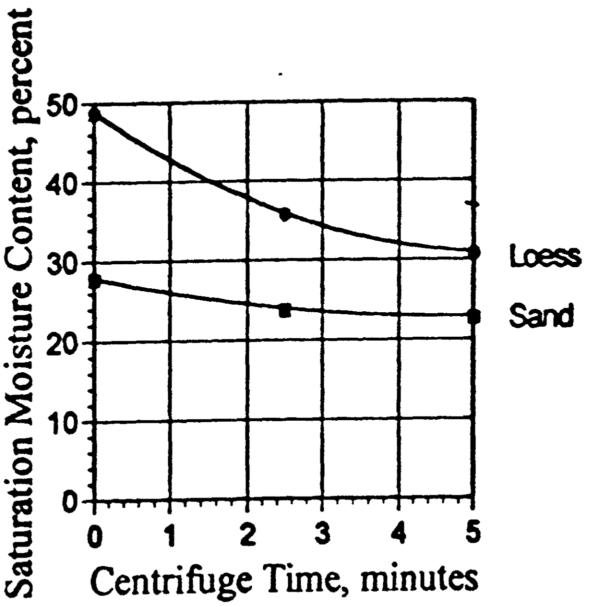


Figure 2. Saturation moisture content of loess and sand as a function of centrifuge time. Loess samples MMV-5, 6, 7, and 9, MMV-18, 19, 20, and 21, and NVP-2 were averaged to give "Loess". Silty sand samples NEC-17, 18, 19, and 20 were averaged to give "Sand". The centrifuge was operated at about 1600xG.

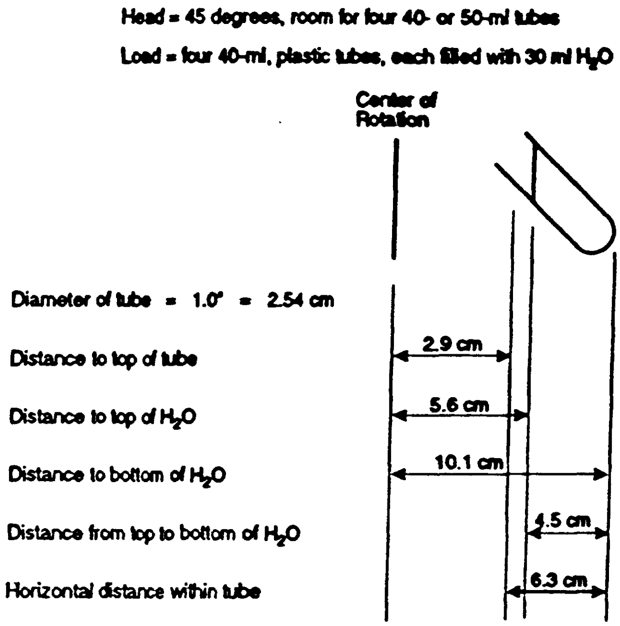


Figure 3. Configuration and dimensions of the IEC (Model CL) clinical centrifuge.

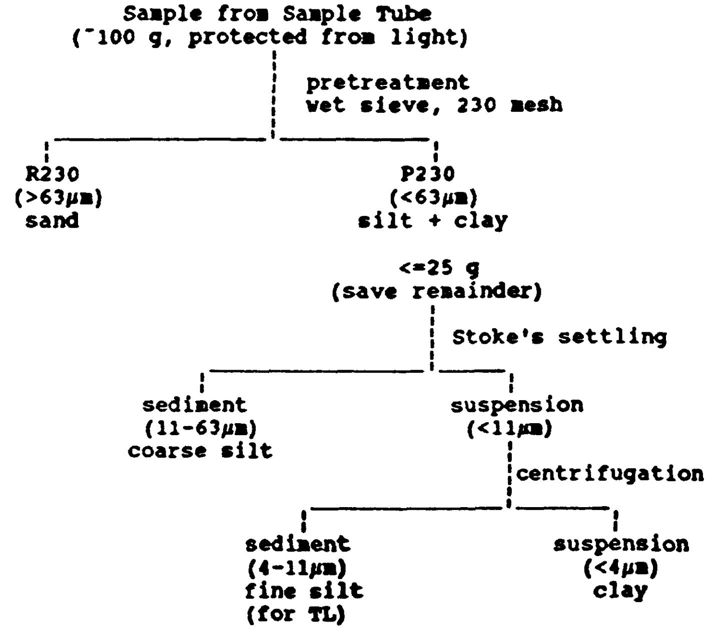
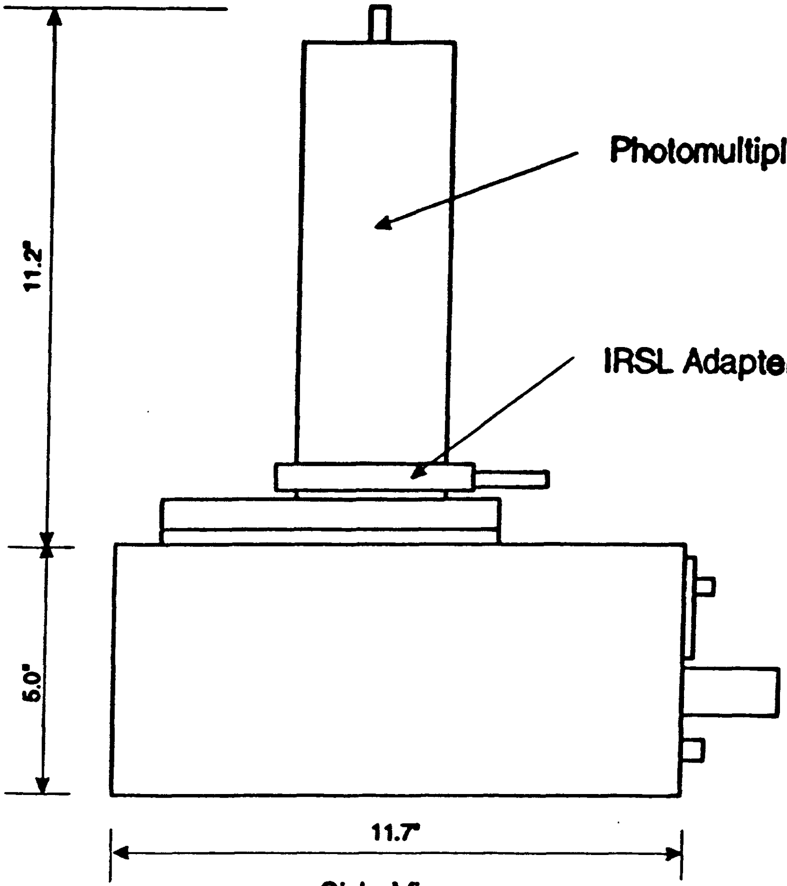
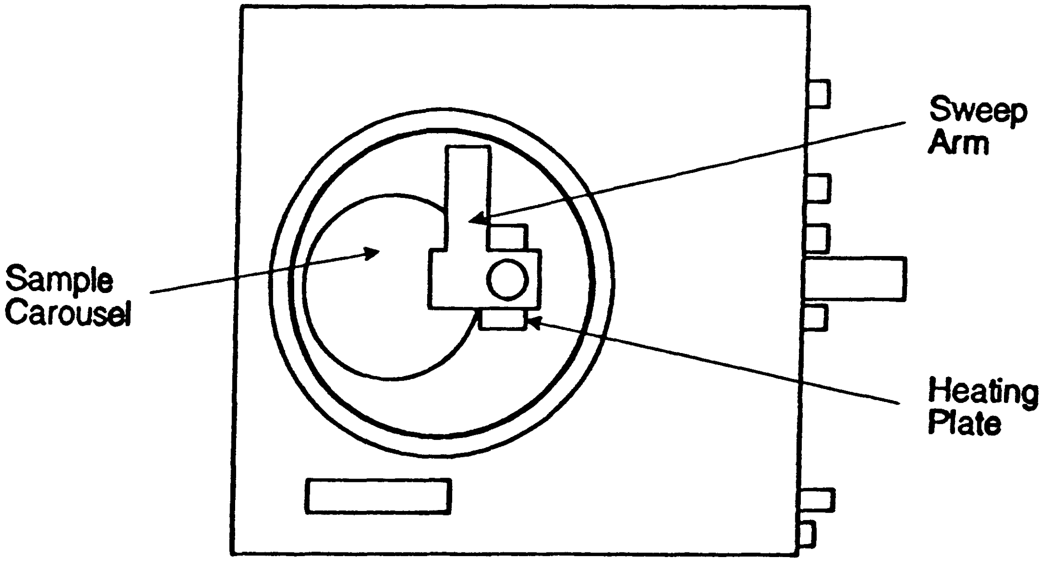


Figure 4. Flow diagram for the sample from the sample tube.



Side View



Top View (Photomultiplier and IRSL adapter removed)

Figure 5. Daybreak Model 1100 TL instrument (adapted from Daybreak Product Description).

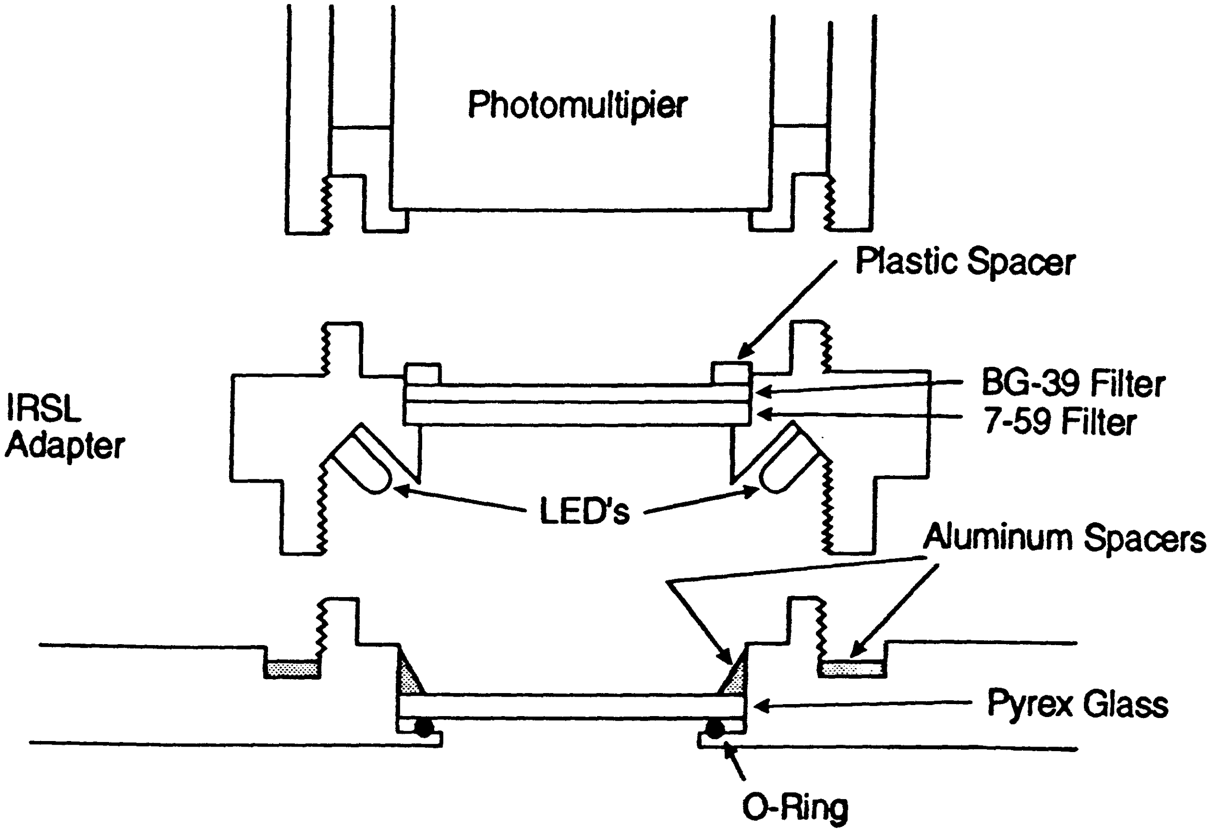


Figure 6. Schematic diagram of Photomultiplier/Filter/IRSL Assembly.

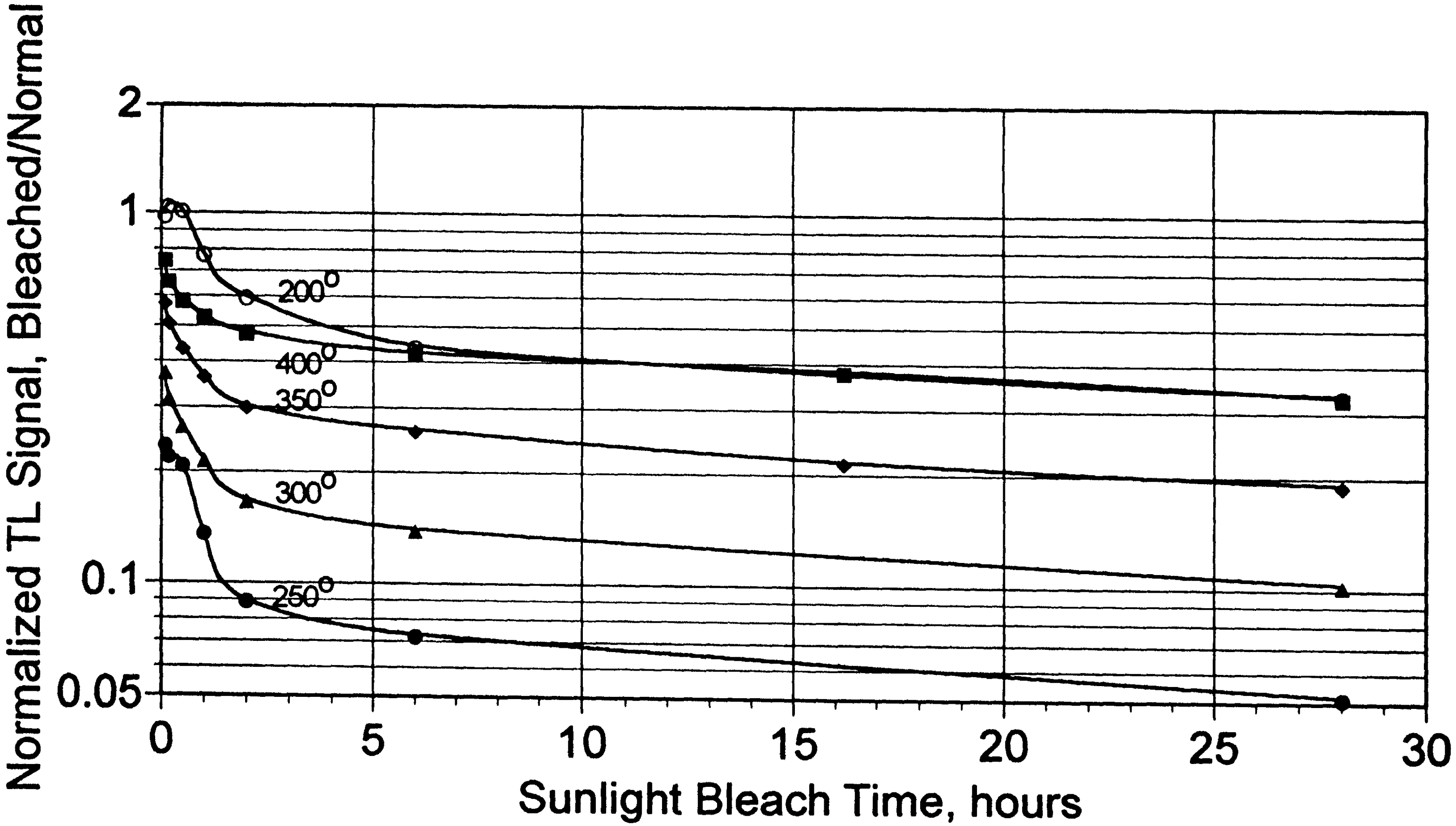


Figure 7. Sunlight bleach curves for sample MMV-5.

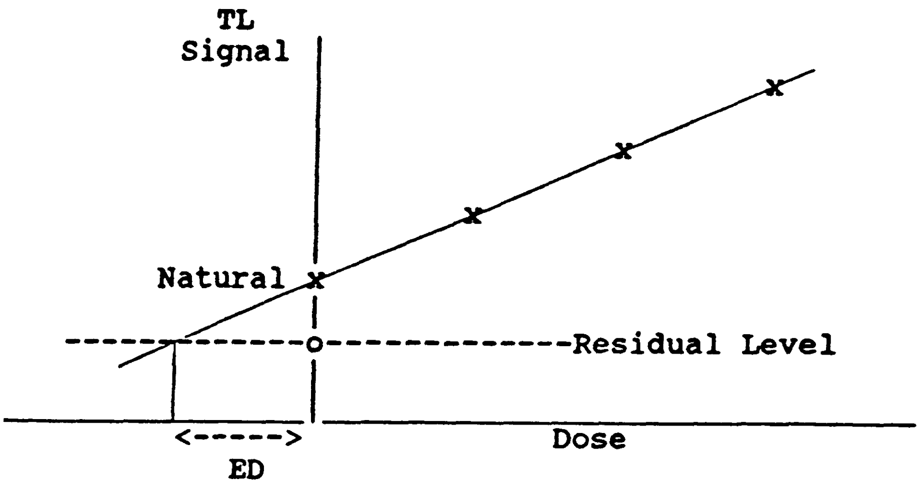


Figure 8. Total bleach TL method.

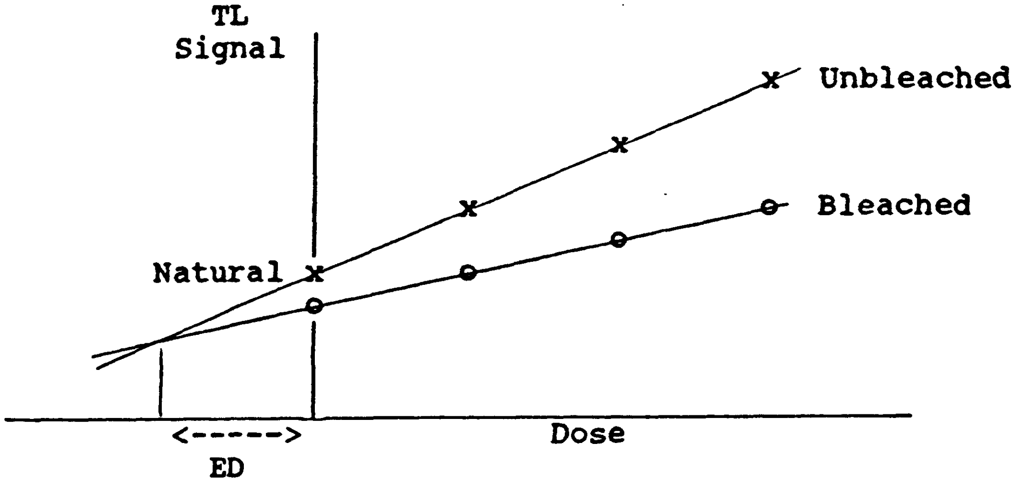


Figure 9. Partial bleach TL method.

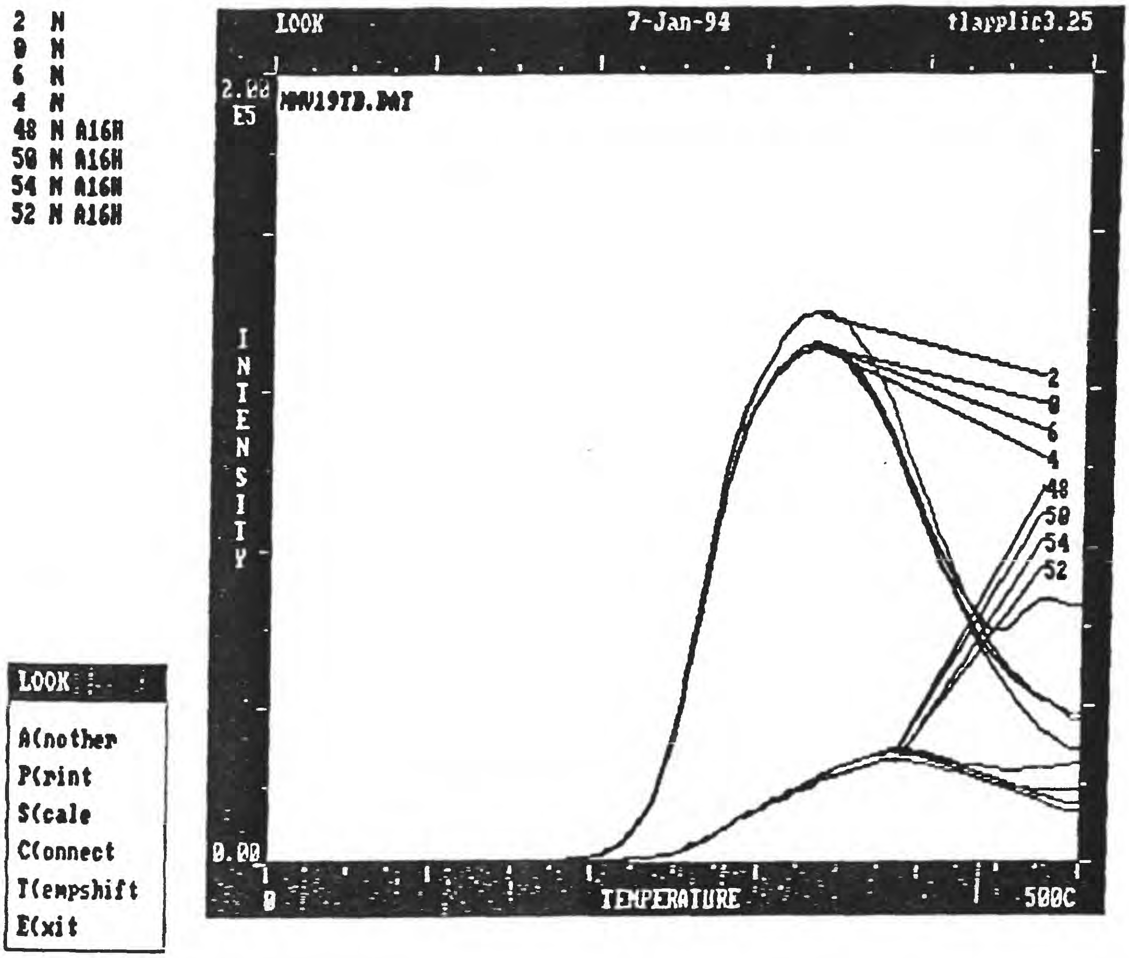


Figure 10. Temperature-shifted and normalized glowcurves for sample MMV-19 (total bleach method).

Runs 0, 2, 4, 6 = natural

Runs 48, 50 , 52, 54 = natural + 16 hour sunlight bleach

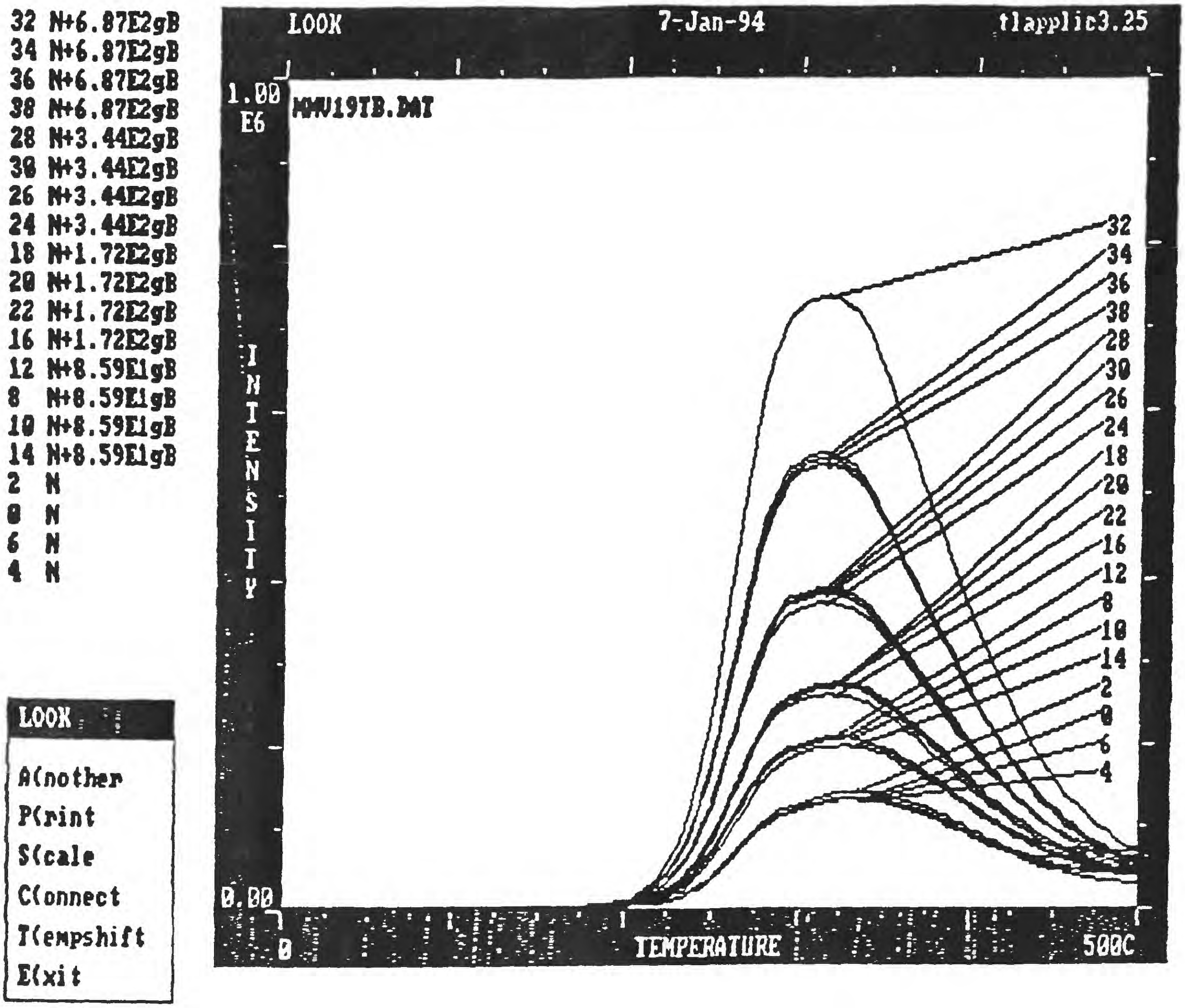


Figure 11. Temperature-shifted and normalized glowcurves for sample MMV-19 (total bleach method). Note poor agreement for disk in run 32 compared to disks in runs 34, 36, and 38.

Runs 0, 2, 4, 6 = natural + 687 Gy

Runs 8, 10, 12, 14 = natural + 344 Gy

Runs 16, 18, 20, 22 = natural + 172 Gy

Runs 24, 26, 28, 30 = natural + 86 Gy

Runs 32, 34, 36, 38 = natural

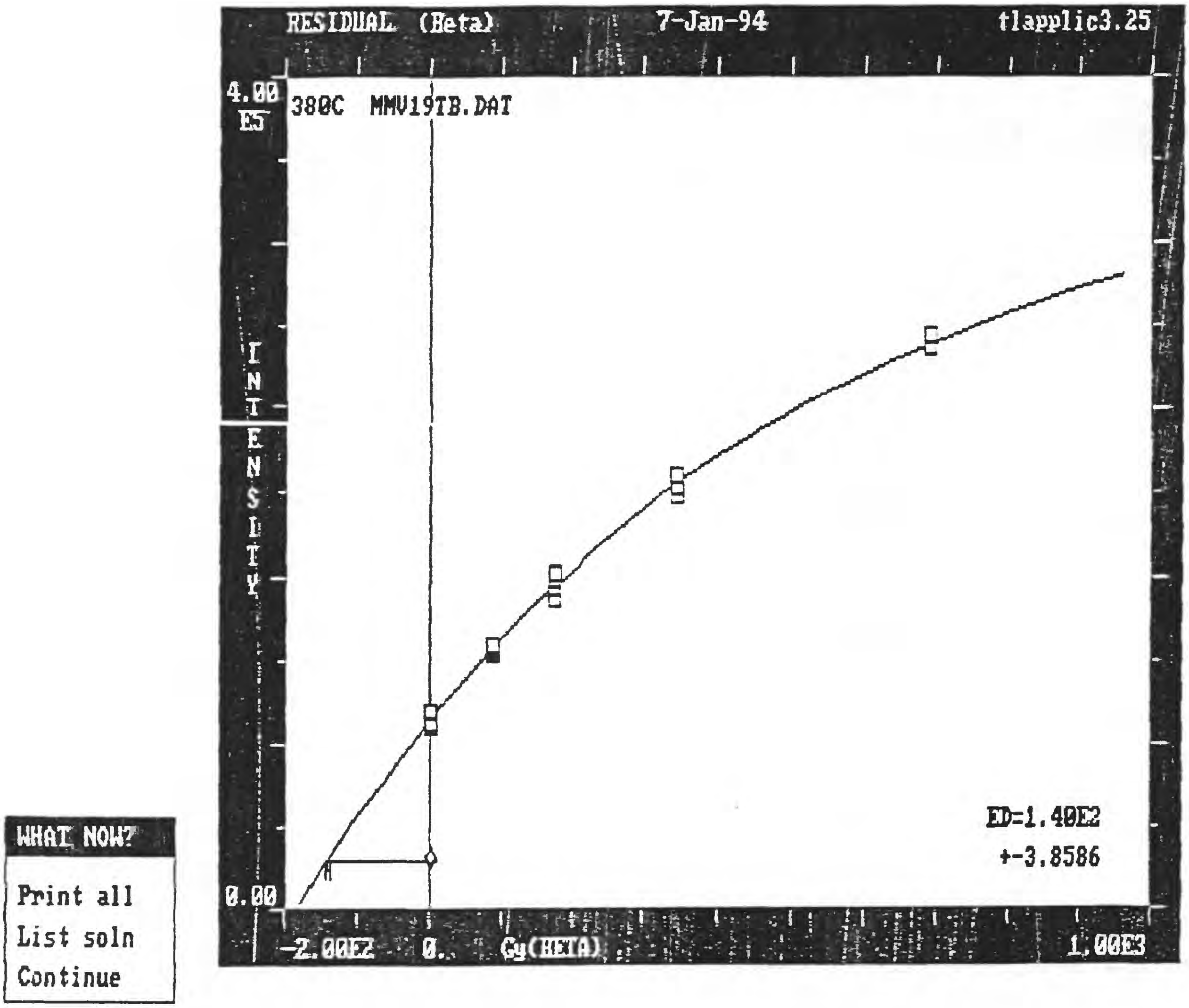


Figure 12. Exponential growth curve fitted to points for 380°C for sample MMV-19 (total bleach method). Run 32 was not included. The intersection between the growth curve and the line through the point for the 16 hour sunlight bleach defines the equivalent dose.

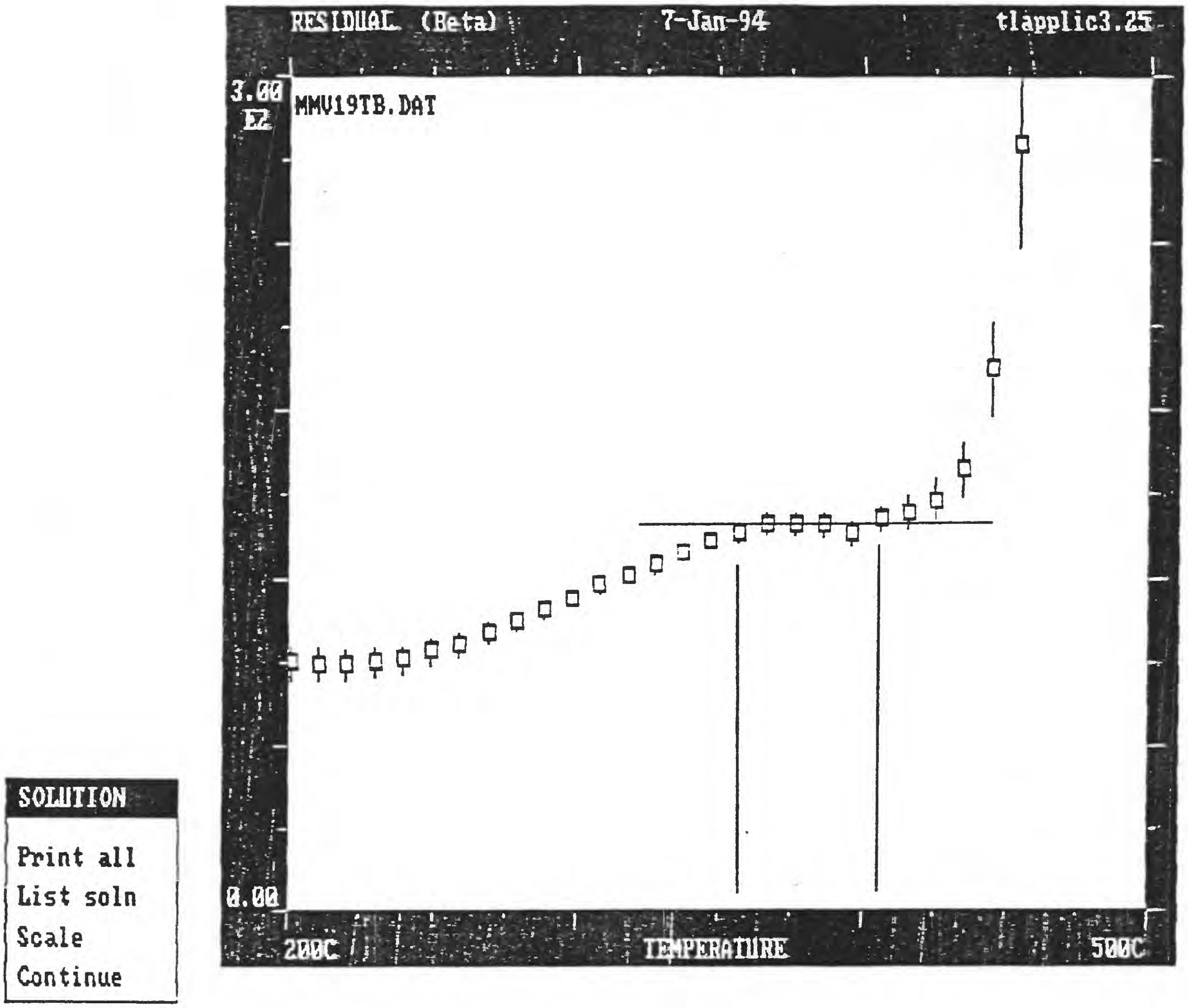


Figure 13. Plot of equivalent dose vs. temperature for sample MMV-19 (total bleach method). Run 32 not included. The plateau occurs from 360° to 410°C.

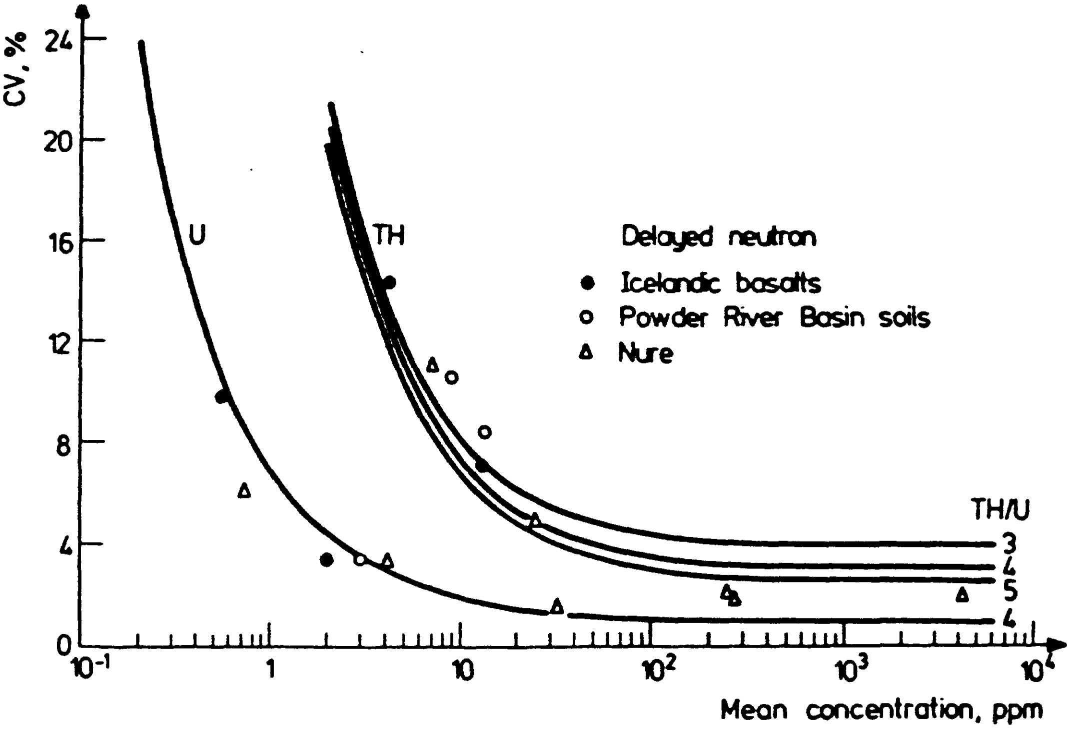


Figure 14. Precision of determinations of U and Th by delayed neutron as a function of concentration as determined from duplicates (from Millard and Keaton, 1982).

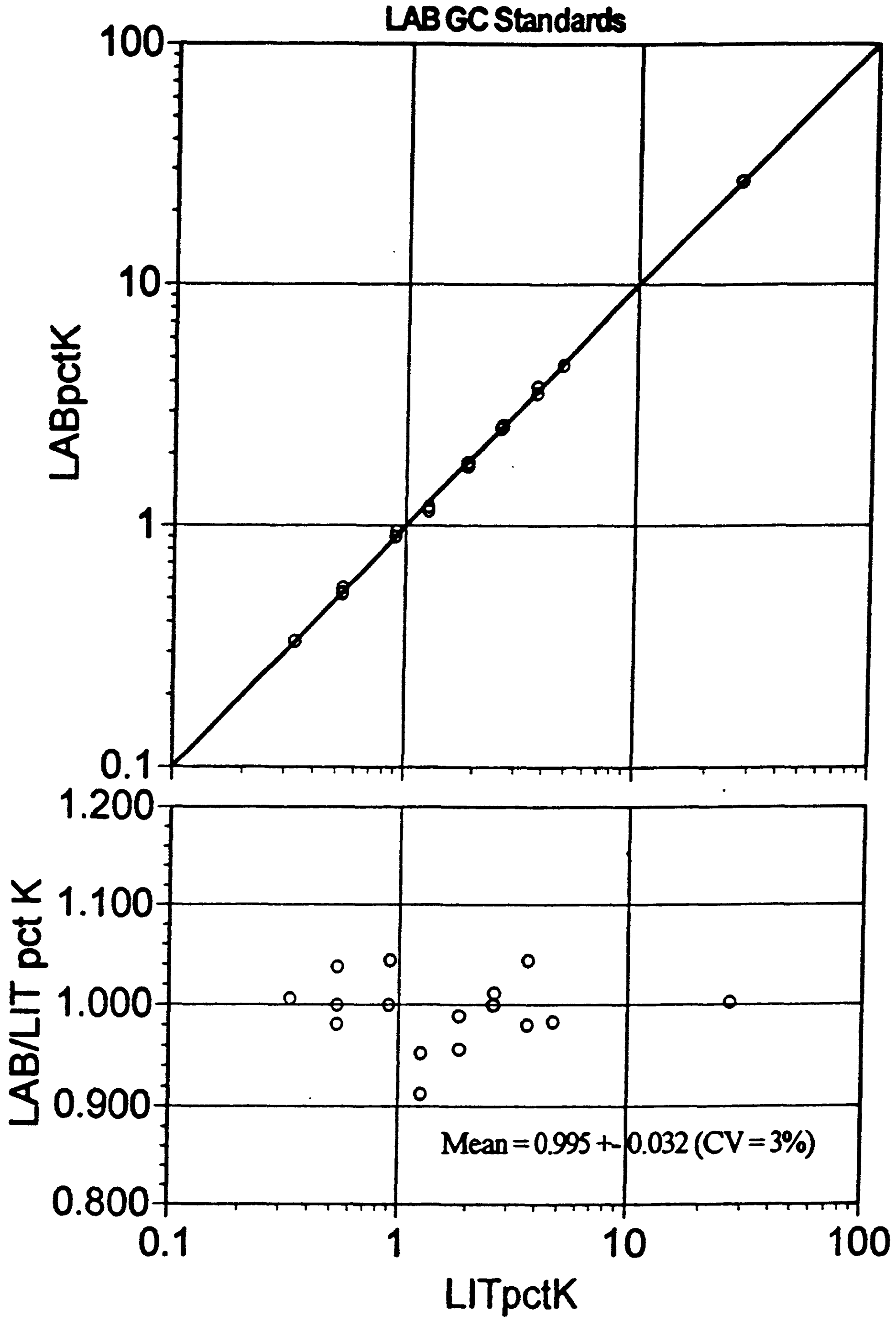


Figure 15. Agreement between laboratory gamma spectrometry and literature values for K.

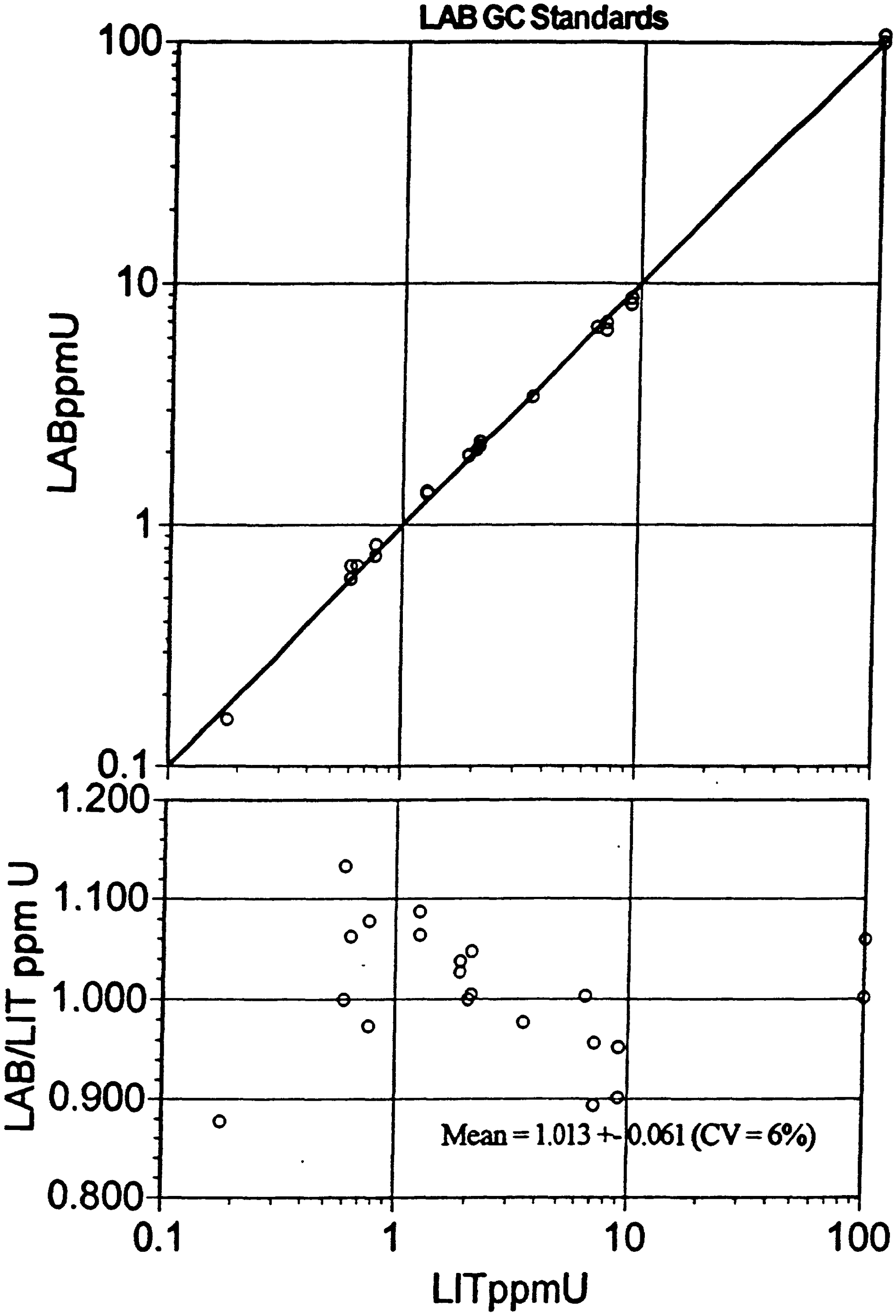


Figure 16. Agreement between laboratory gamma spectrometry and literature values for U.

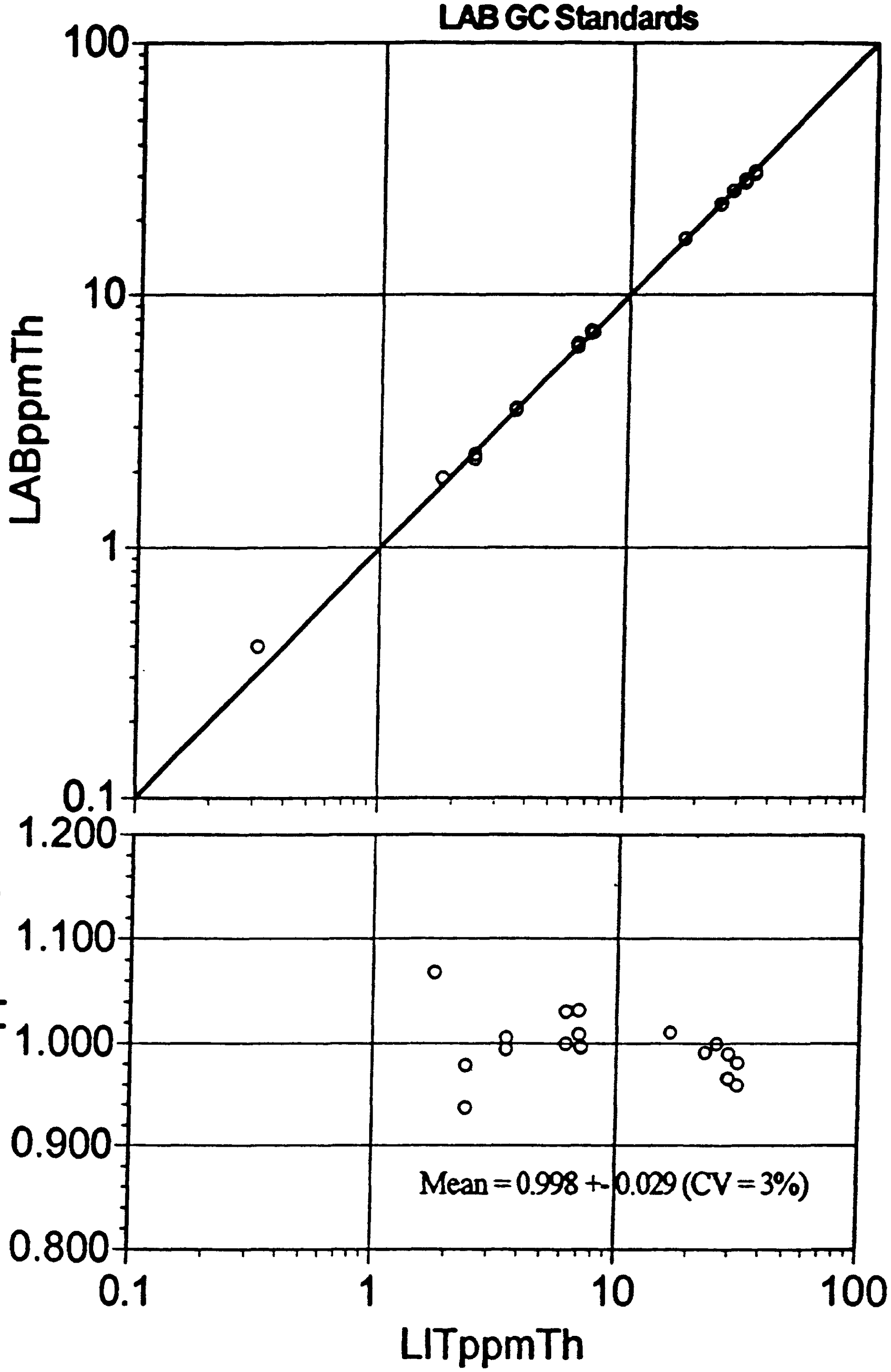


Figure 17. Agreement between laboratory gamma spectrometry and literature values for Th.

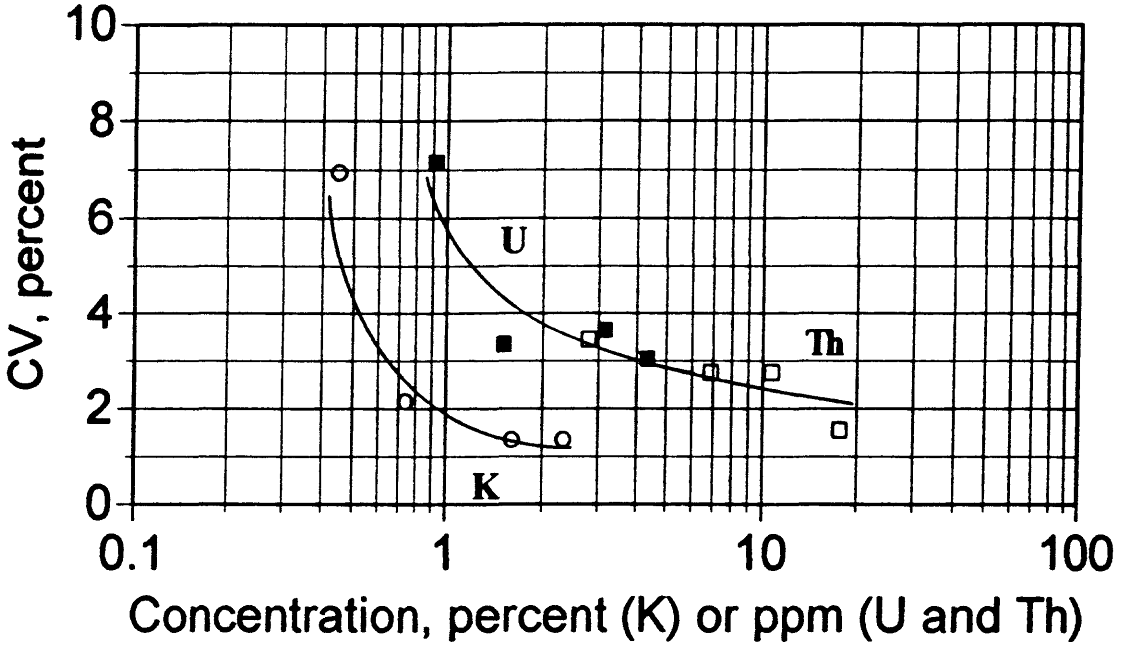


Figure 18. Precision of field gamma spectrometry values for K, U, and Th as a function of concentration as determined from duplicates.

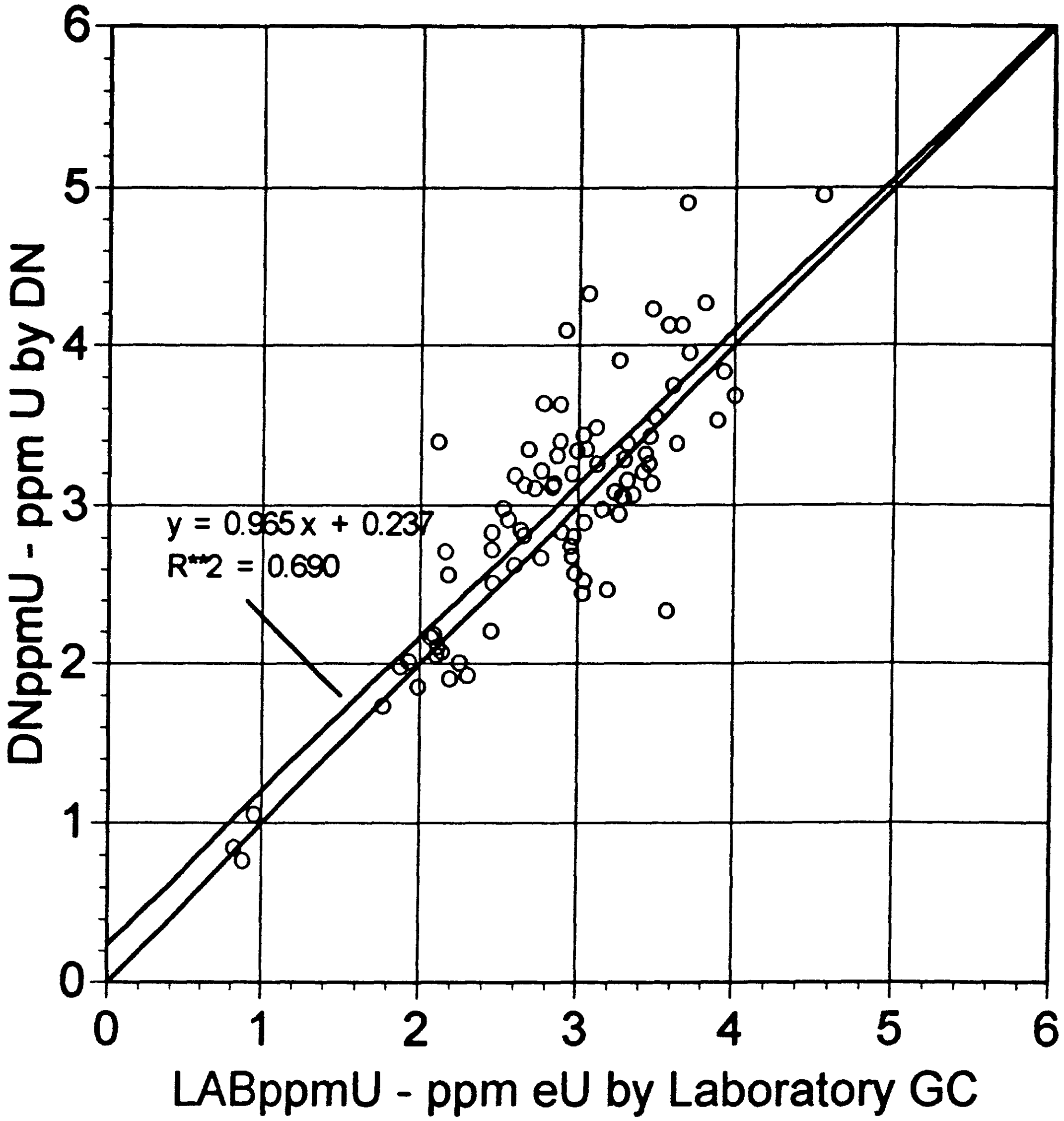


Figure 19. Agreement between delayed neutron and laboratory gamma spectrometry values for U.

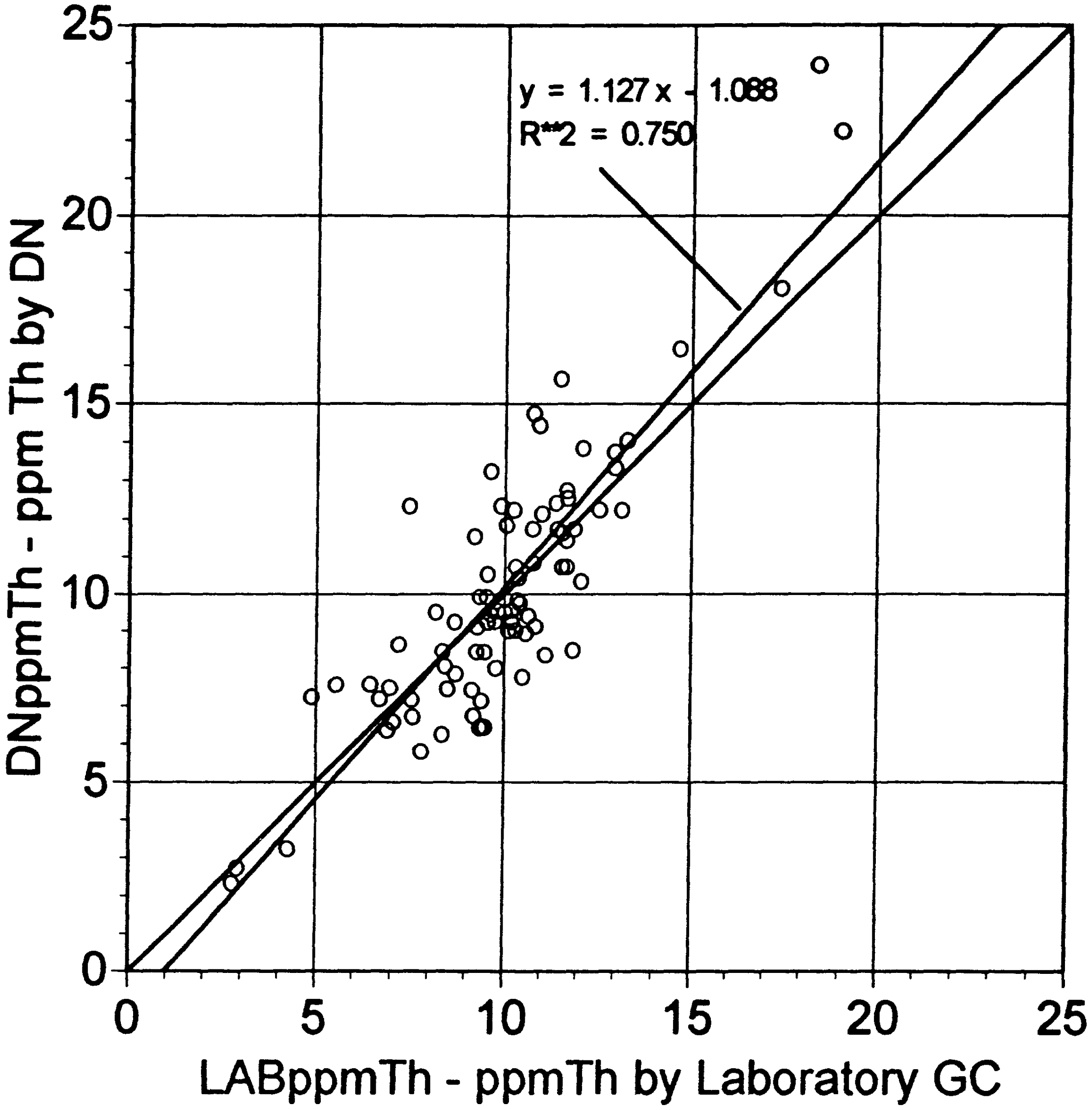


Figure 20. Agreement between delayed neutron and laboratory gamma spectrometry values for Th.

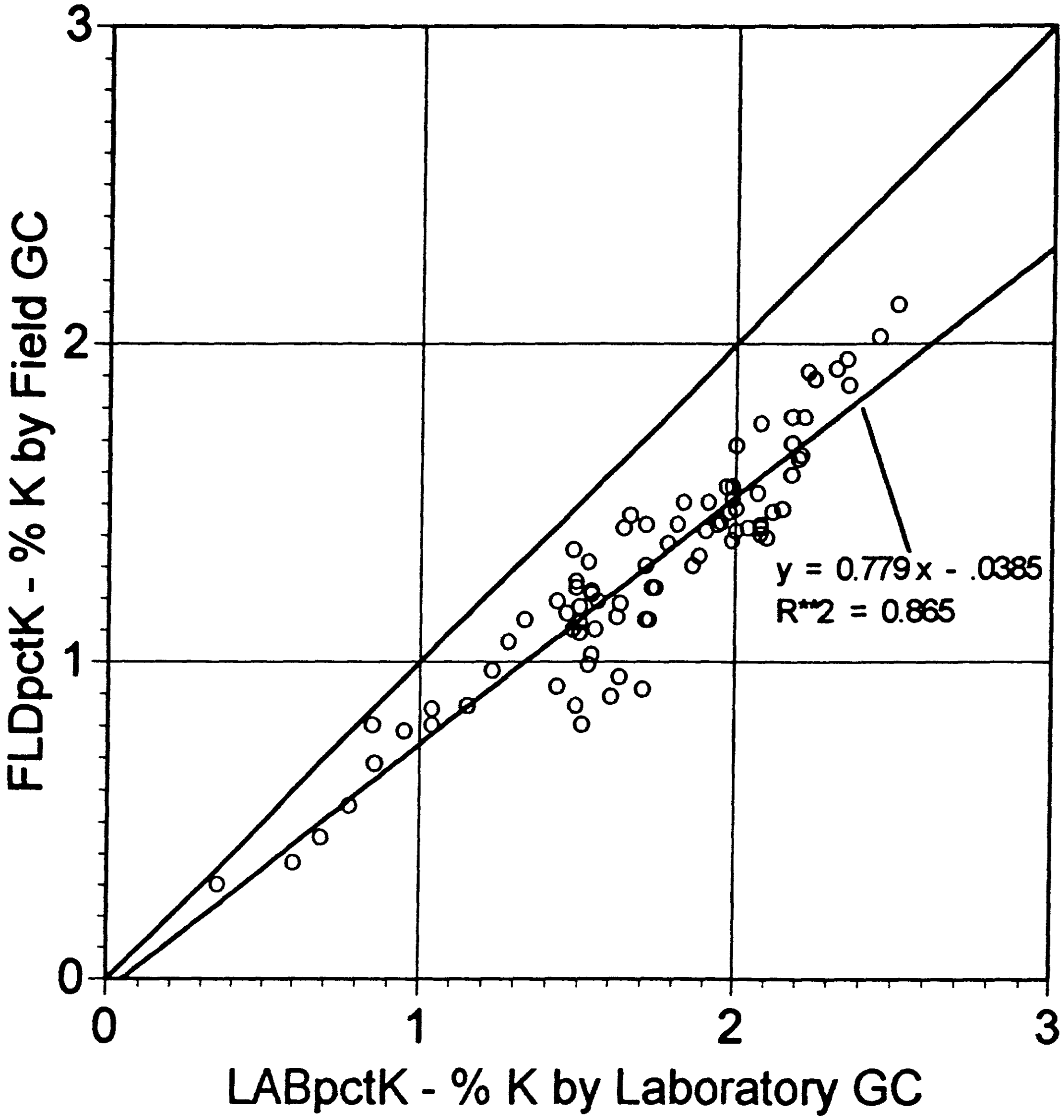


Figure 21. Agreement between field gamma spectrometry and laboratory gamma spectrometry values for K. No correction for moisture.

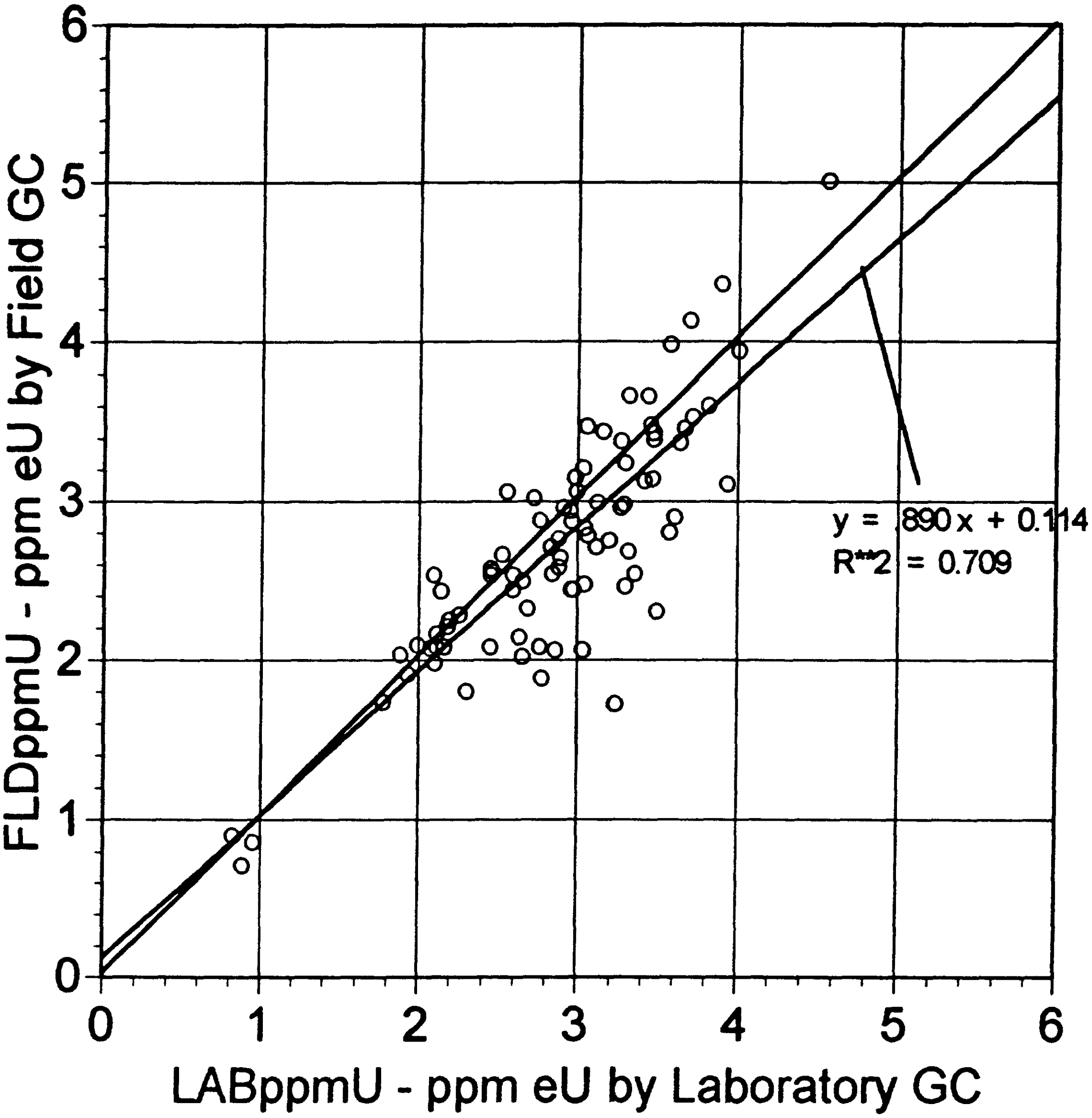


Figure 22. Agreement between field gamma spectrometry and laboratory gamma spectrometry values for U. No correction for moisture.

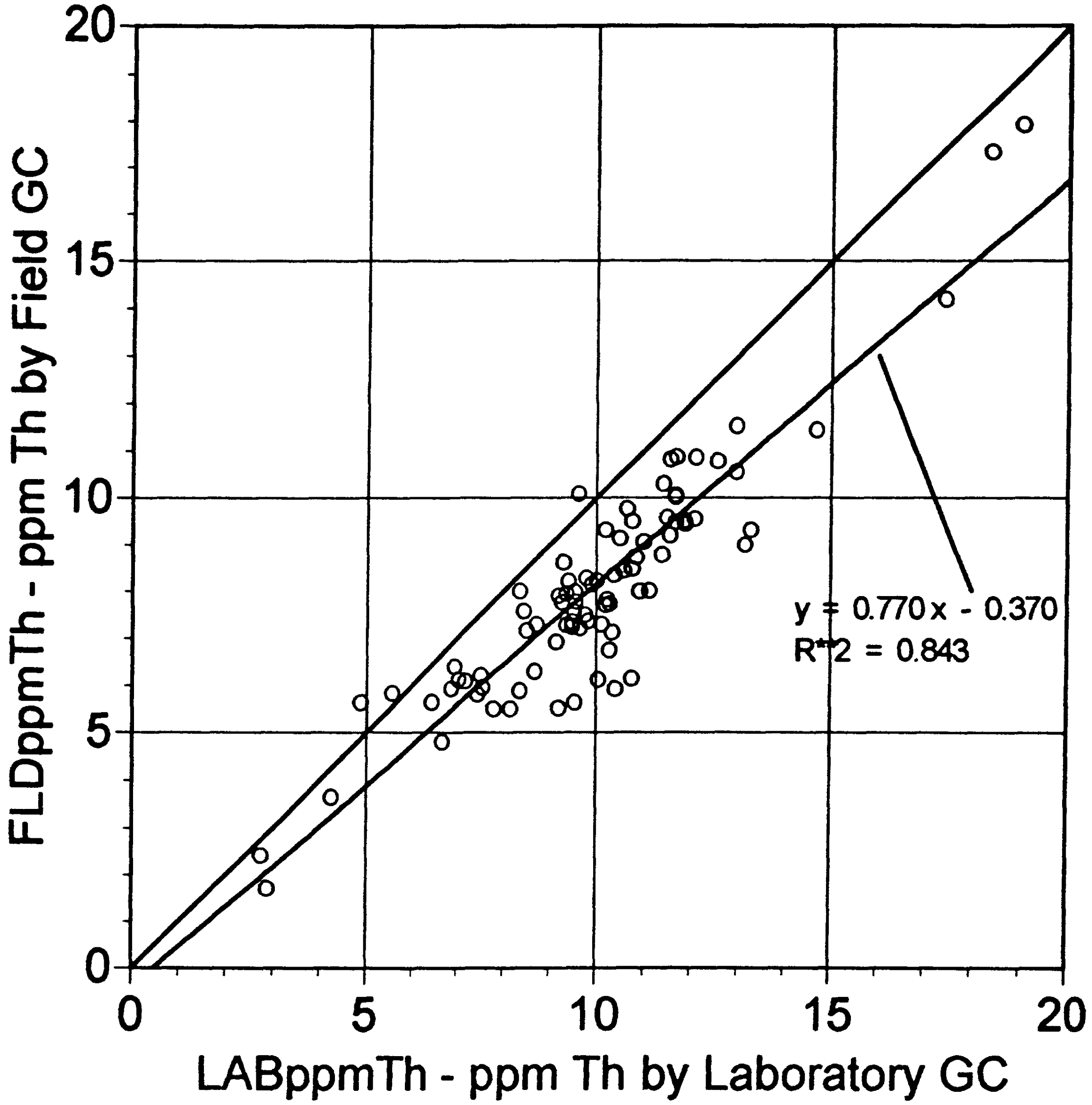


Figure 23. Agreement between field gamma spectrometry and laboratory gamma spectrometry values for Th. No correction for moisture.

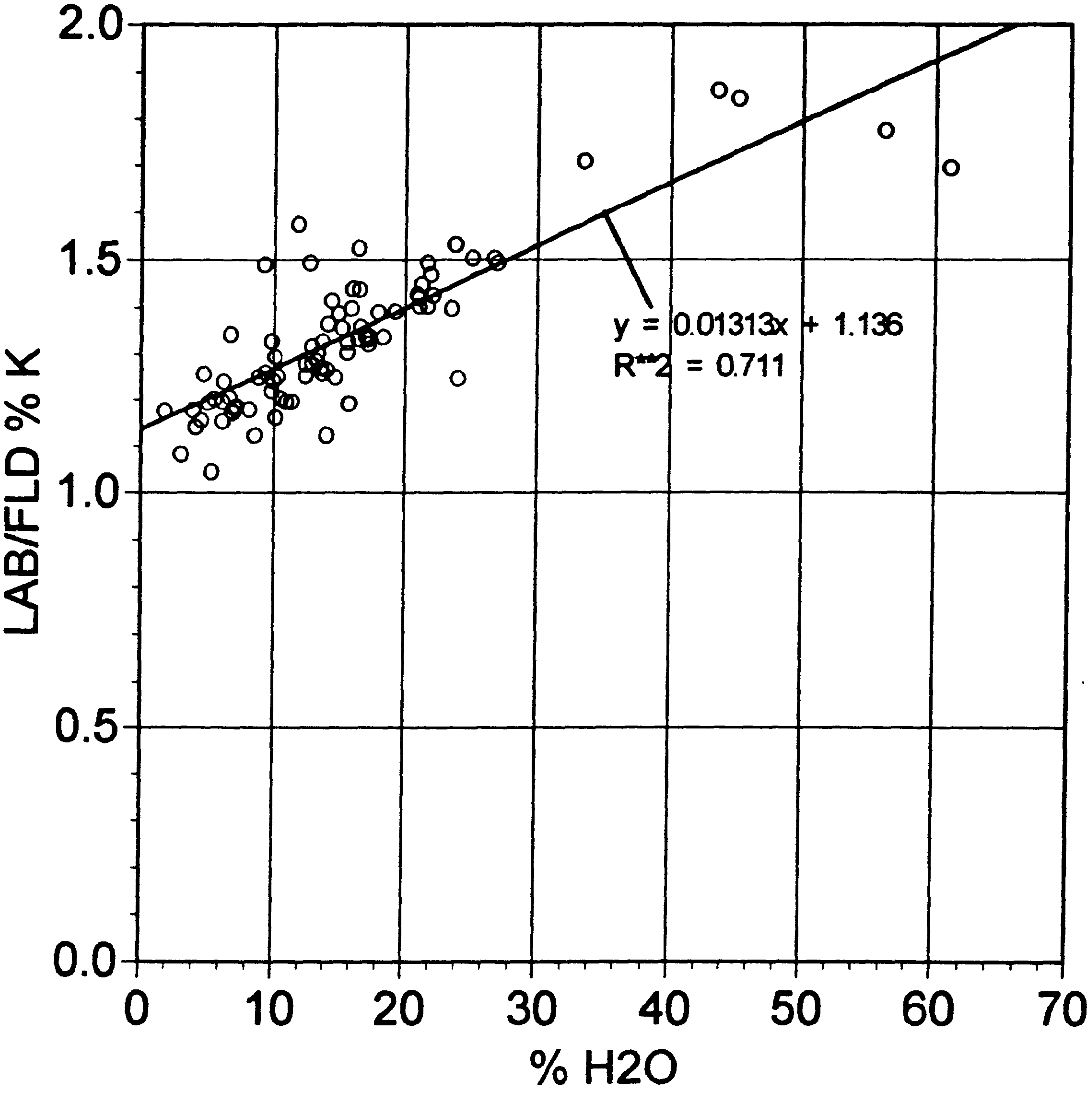


Figure 24. Ratio of laboratory gamma spectrometry values for K to field gamma spectrometry values as a function of the moisture content.

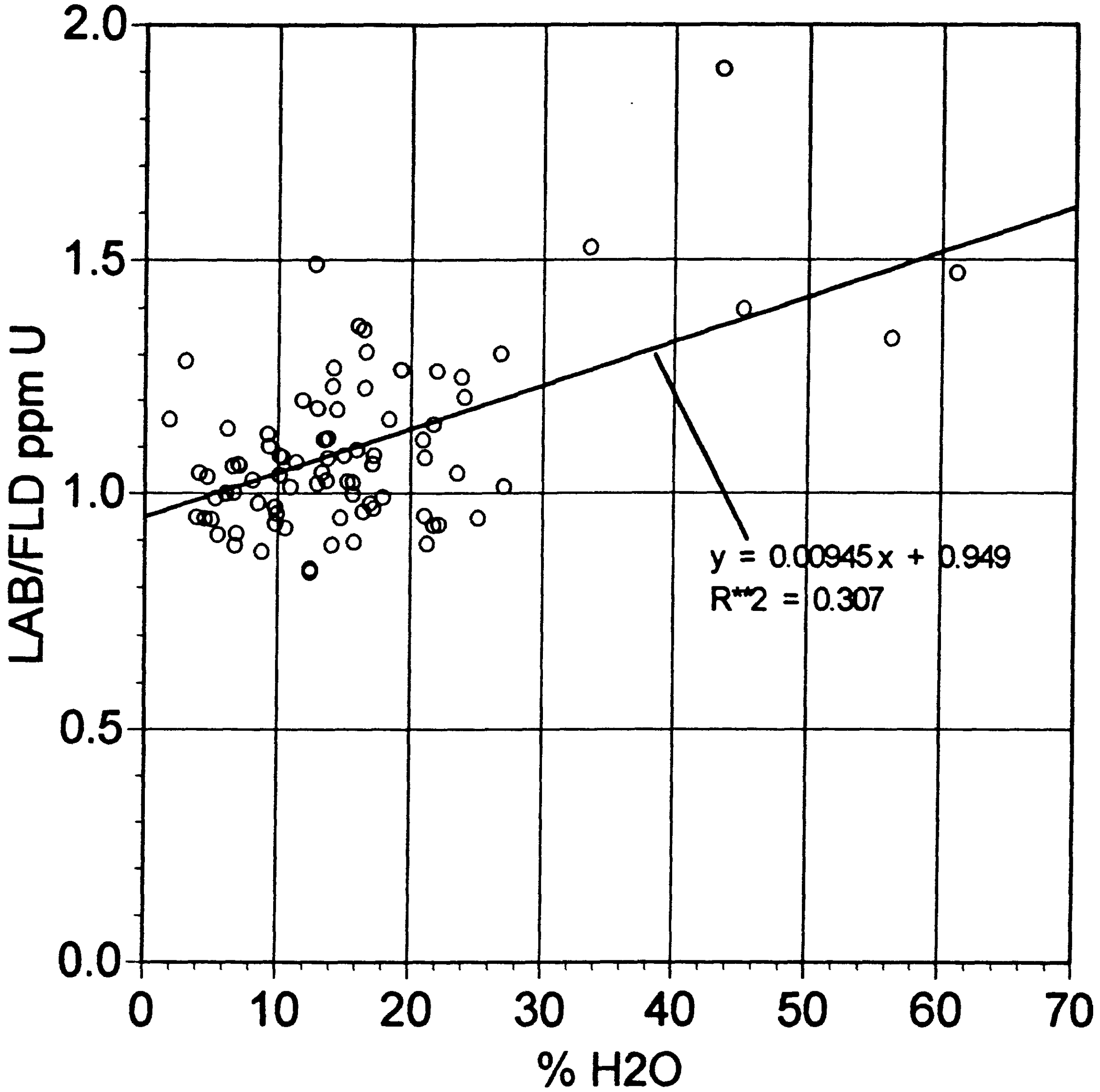


Figure 25. Ratio of laboratory gamma spectrometry values for U to field gamma spectrometry values as a function of the moisture content.

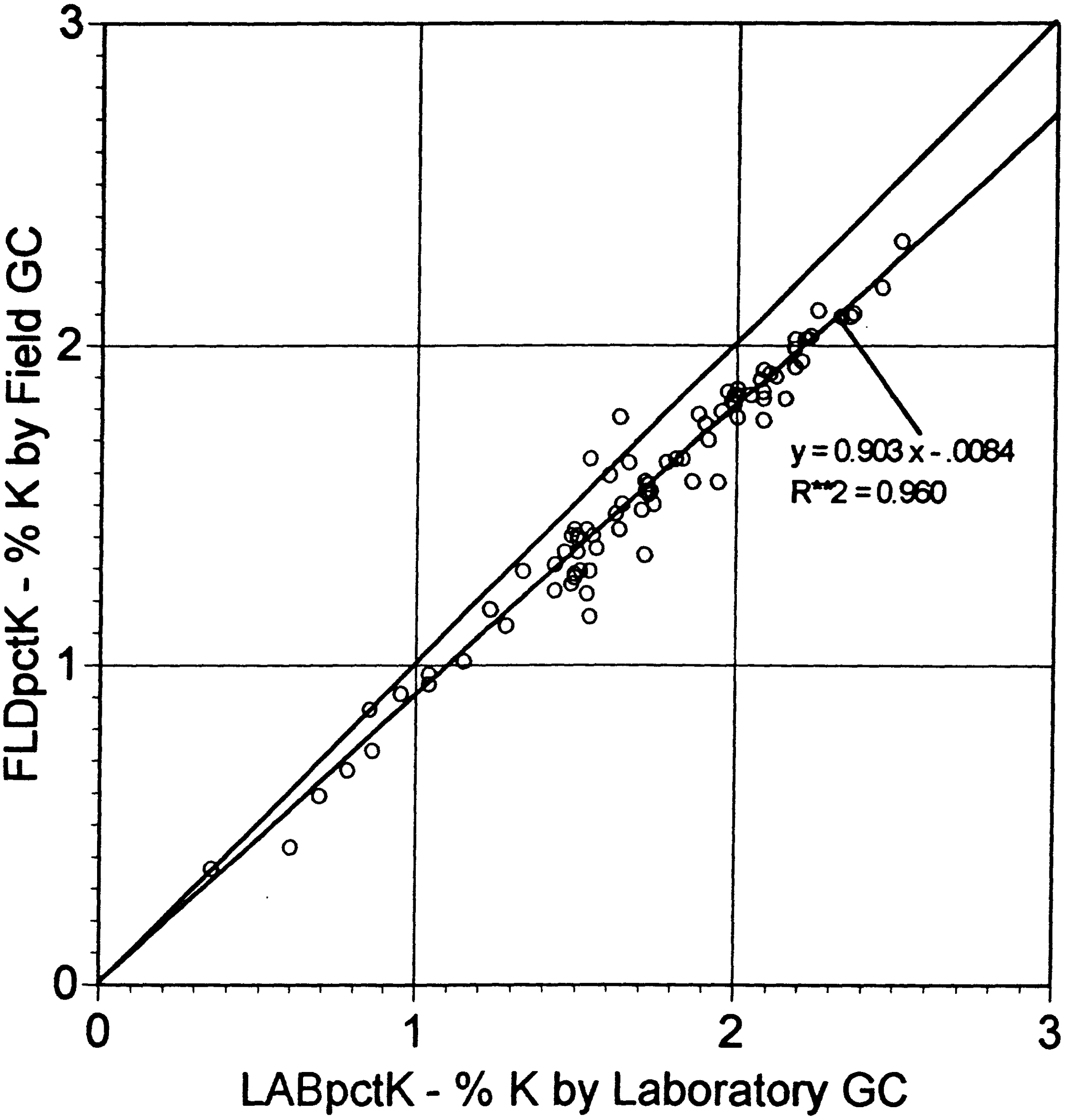


Figure 27. Agreement between field gamma spectrometry and laboratory gamma spectrometry values for K. Field values corrected for moisture content.

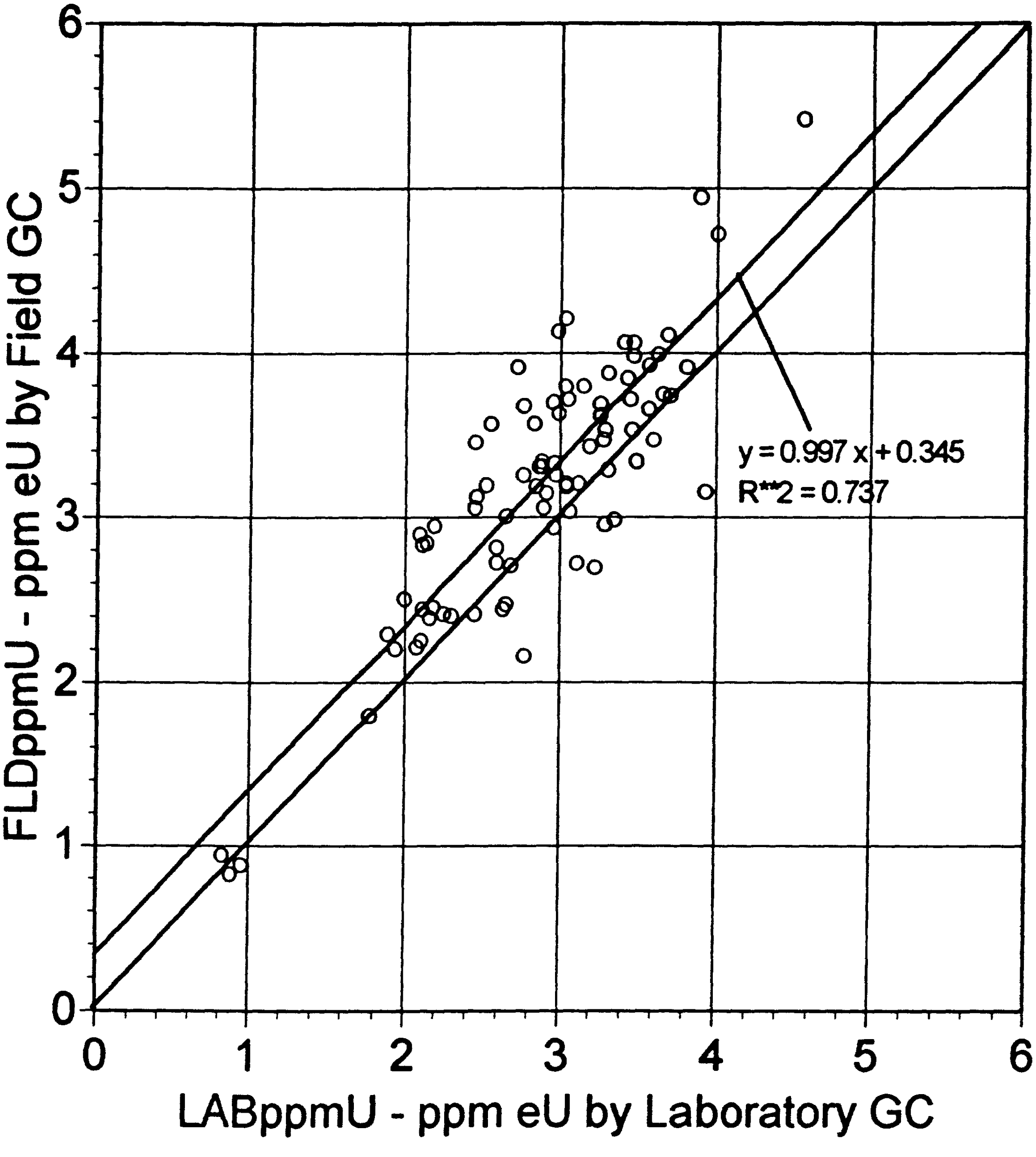


Figure 28. Agreement between field gamma spectrometry and laboratory gamma spectrometry values for U. Field values corrected for moisture content.

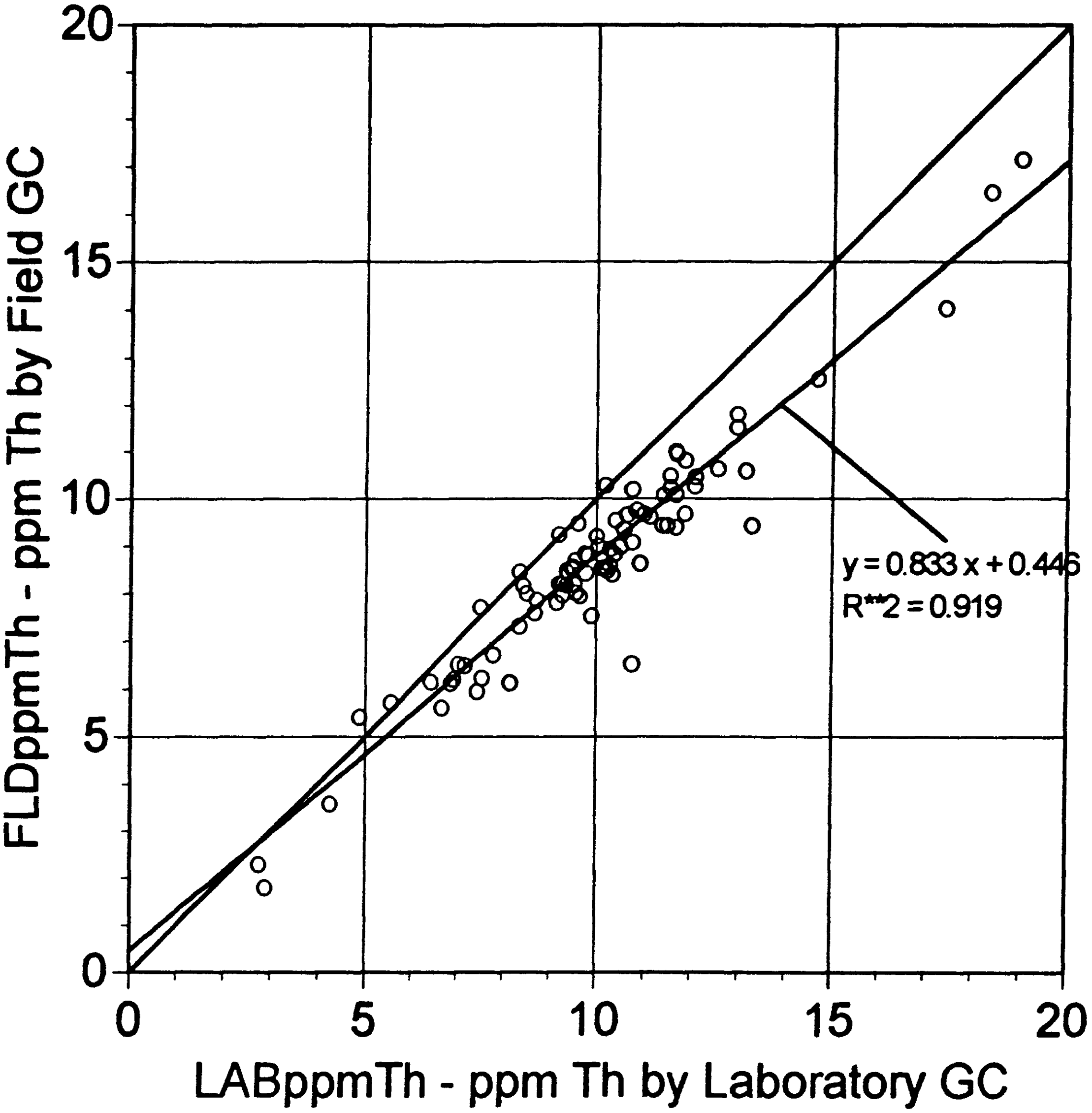


Figure 29. Agreement between field gamma spectrometry and laboratory gamma spectrometry for Th. Field values corrected for moisture content.

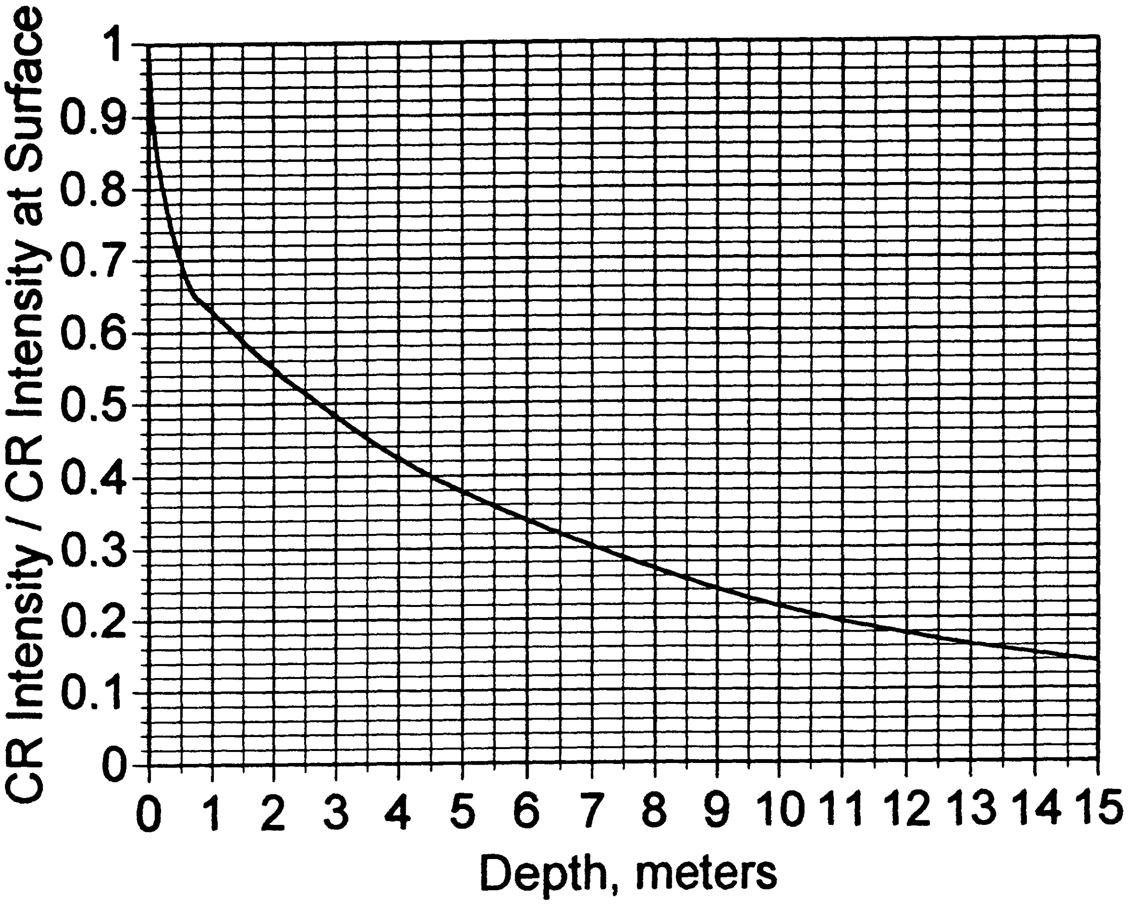


Figure 30. Ratio of cosmic ray intensity to intensity at the surface as a function of depth in sedimentary rock. Density $= 2\ g/cm^3$. Data from Prescott and Hutton (1988).

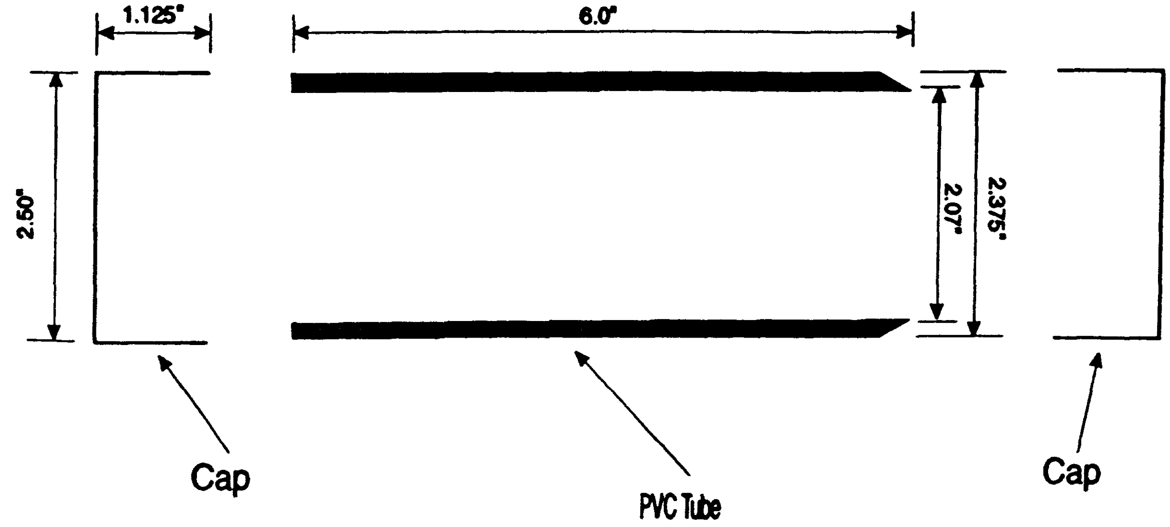


Figure 31. Sample tube.

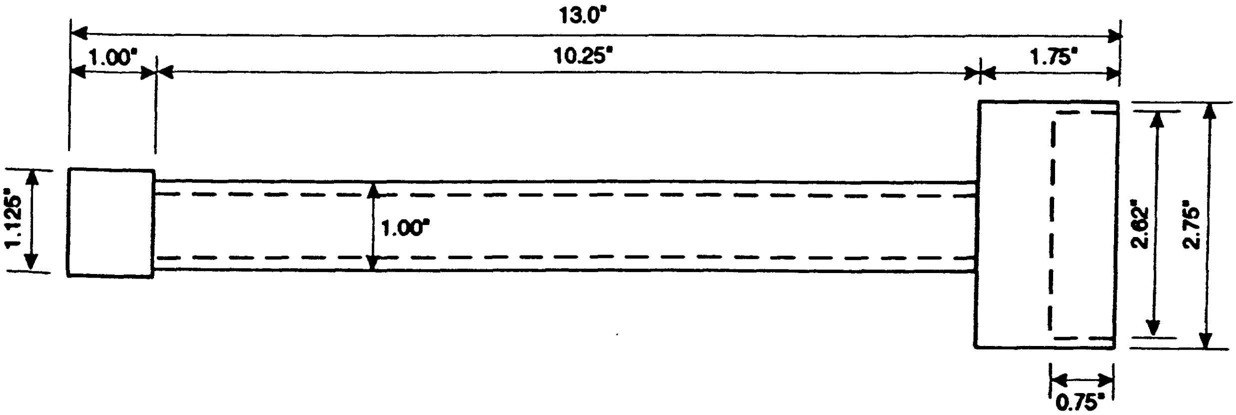
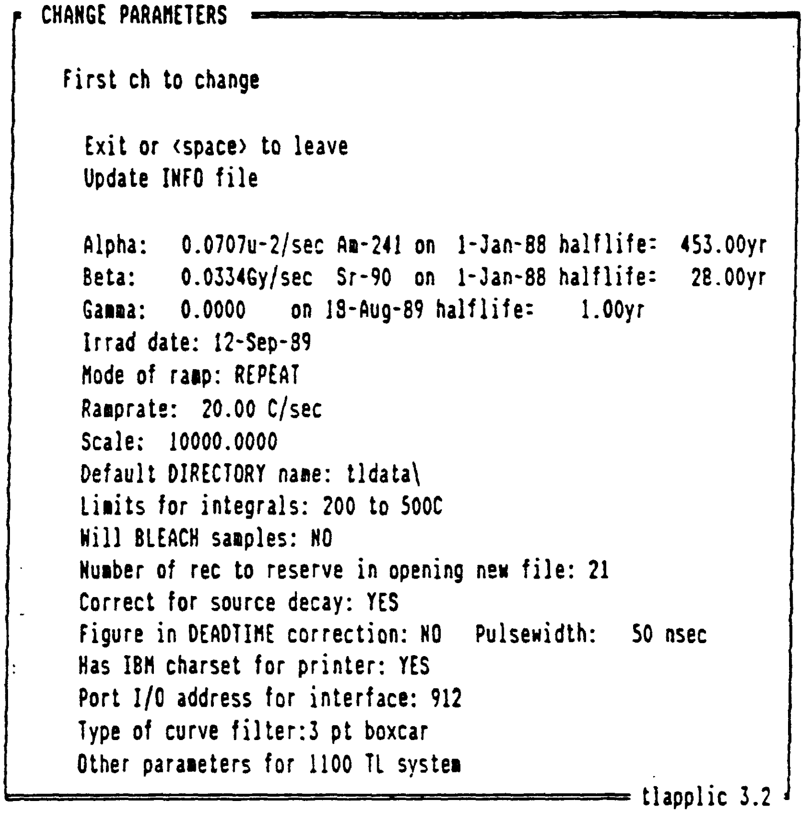
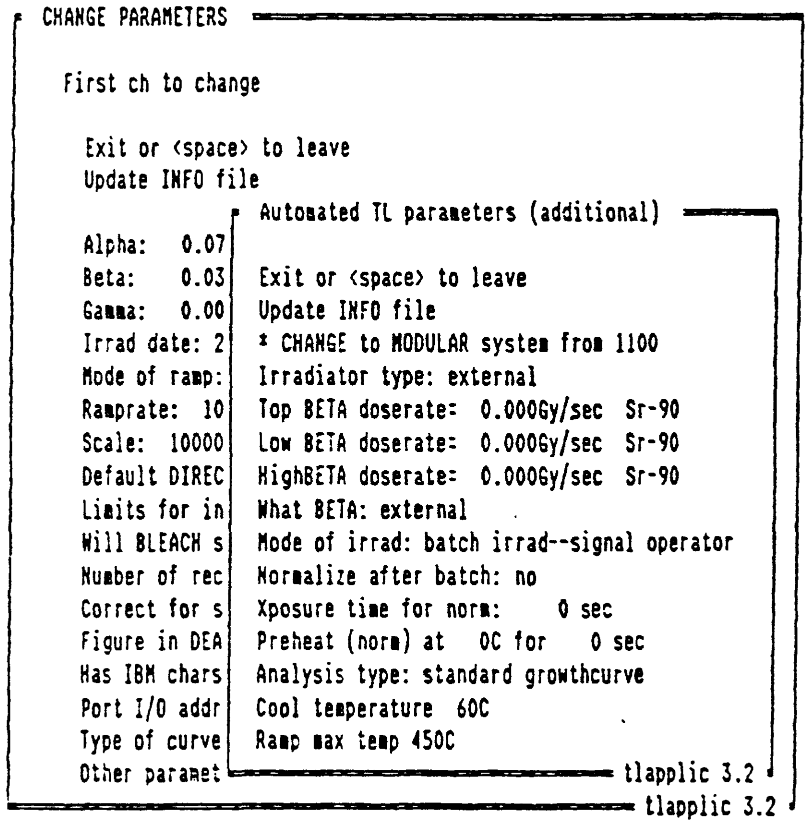


Figure 32. Drive tube.



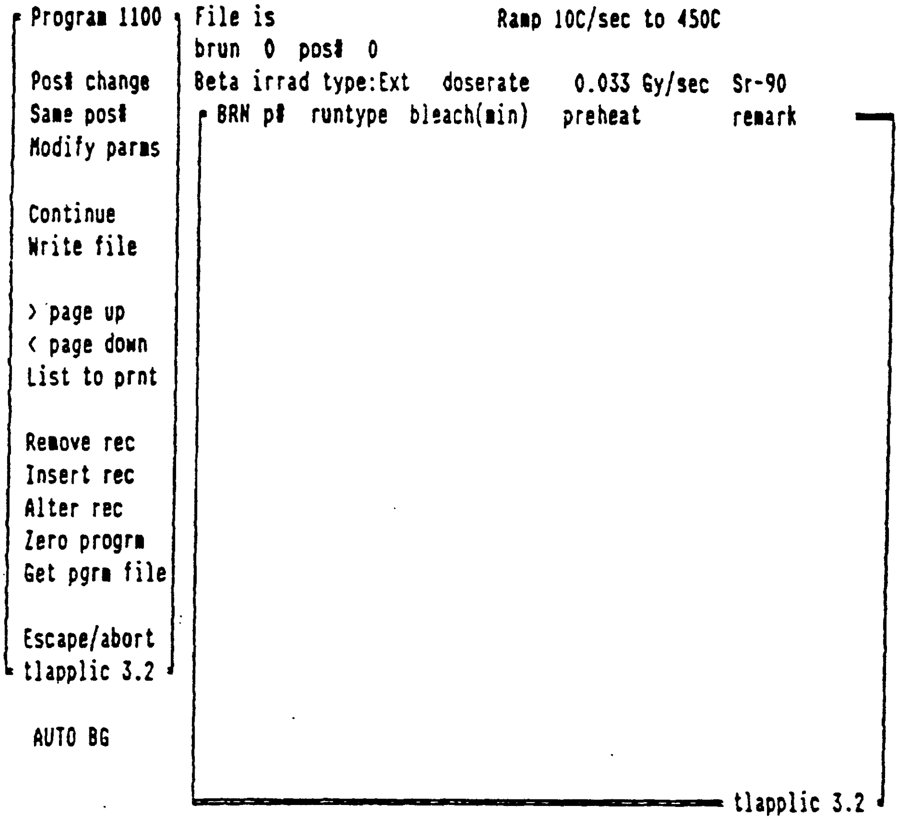
```
CHANGE PARAMETERS

  First ch to change

    Exit or <space> to leave
    Update INFO file

    Alpha:   0.0707u-2/sec Am-241 on  1-Jan-88 halflife:  453.00yr
    Beta:    0.0334Gy/sec  Sr-90  on  1-Jan-88 halflife:   28.00yr
    Gamma:   0.0000     on 18-Aug-89 halflife:    1.00yr
    Irrad date: 12-Sep-89
    Mode of ramp: REPEAT
    Ramprate:  20.00 C/sec
    Scale:  10000.0000
    Default DIRECTORY name: tldata\
    Limits for integrals: 200 to 500C
    Will BLEACH samples: NO
    Number of rec to reserve in opening new file: 21
    Correct for source decay: YES
    Figure in DEADTIME correction: NO   Pulsewidth:   50 nsec
    Has IBM charset for printer: YES
    Port I/O address for interface: 912
    Type of curve filter:3 pt boxcar
    Other parameters for 1100 TL system
                                                       tlapplic 3.2
```

Figure 33. Change Parameters menu (Bortelot, 1990, figure 1).



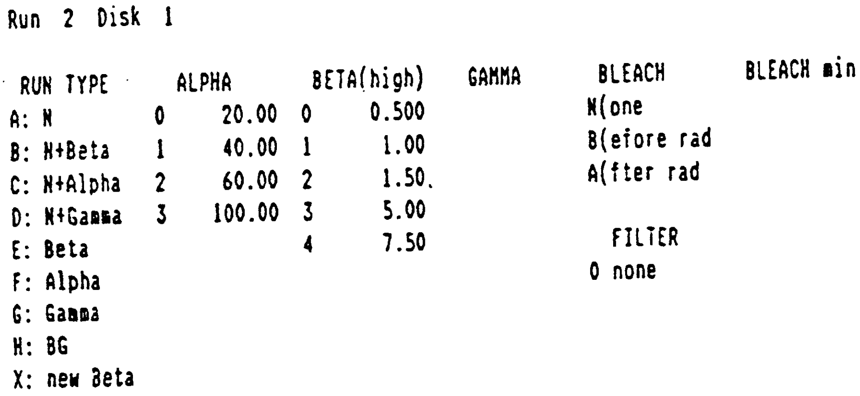
```
CHANGE PARAMETERS

  First ch to change

    Exit or <space> to leave
    Update INFO file
                     Automated TL parameters (additional)
    Alpha:  0.07
    Beta:   0.03     Exit or <space> to leave
    Gamma:  0.00     Update INFO file
    Irrad date: 2    * CHANGE to MODULAR system from 1100
    Mode of ramp:    Irradiator type: external
    Ramprate:  10    Top BETA doserate:  0.000Gy/sec  Sr-90
    Scale:  10000    Low BETA doserate:  0.000Gy/sec  Sr-90
    Default DIREC    HighBETA doserate:  0.000Gy/sec  Sr-90
    Limits for in    What BETA: external
    Will BLEACH s    Mode of irrad: batch irrad--signal operator
    Number of rec    Normalize after batch: no
    Correct for s    Xposure time for norm:     0 sec
    Figure in DEA    Preheat (norm) at   0C for   0 sec
    Has IBM chars    Analysis type: standard growthcurve
    Port I/O addr    Cool temperature  60C
    Type of curve    Ramp max temp 450C
    Other paramet                                tlapplic 3.2
                                                   tlapplic 3.2
```

Figure 34. Automated TL Parameters menu (Bortelot, 1990, figure 2).



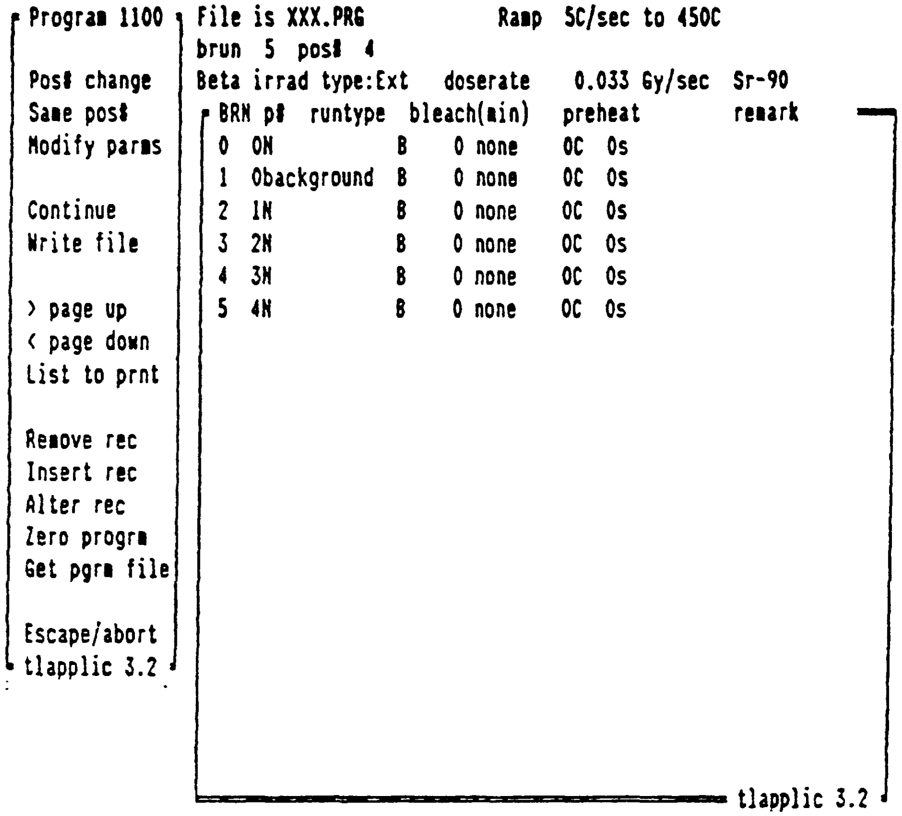
```
Program 1100   File is                 Ramp 10C/sec to 450C
               brun  0  pos#  0
Pos# change    Beta irrad type:Ext  doserate   0.033 Gy/sec  Sr-90
Same pos#       BRN p#  runtype  bleach(min)  preheat        remark
Modify params

Continue
Write file

> page up
< page down
List to prnt

Remove rec
Insert rec
Alter rec
Zero progrm
Get pgrm file

Escape/abort
tlapplic 3.2

AUTO BG
                                                          tlapplic 3.2
```

Figure 35. Batch program display screen before any batch runs have been entered (Bortelot, 1990, figure 8).



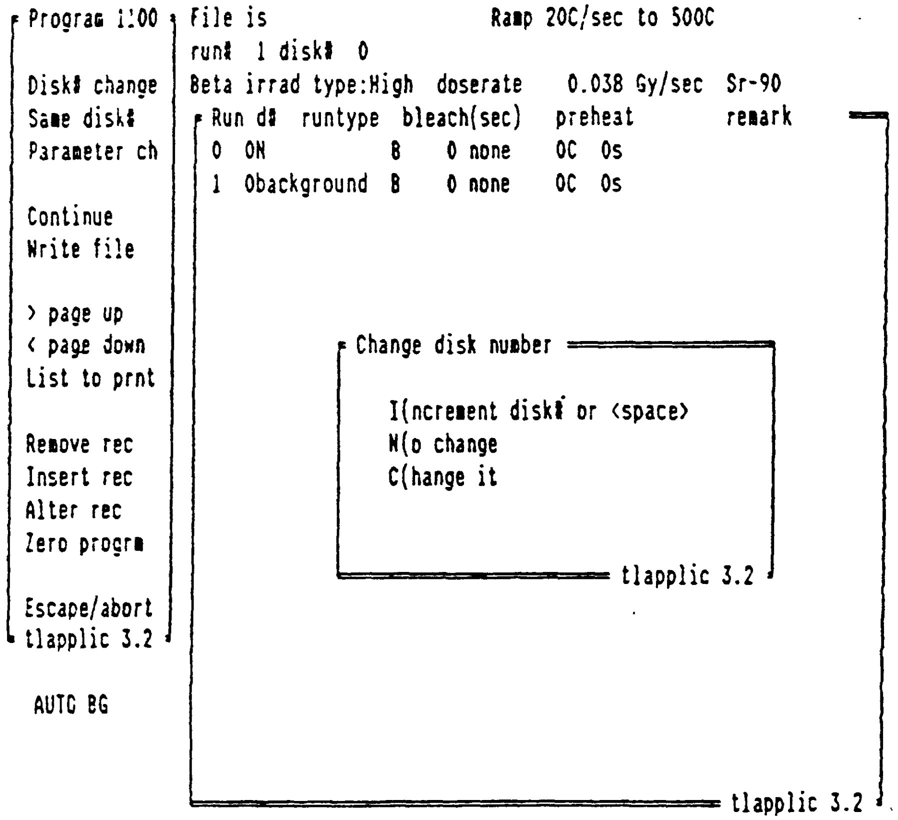
```
Run  2  Disk  1

 RUN TYPE      ALPHA        BETA(high)   GAMMA     BLEACH       BLEACH min
A: N          0     20.00  0      0.500            N(one
B: N+Beta     1     40.00  1       1.00            B(efore rad
C: N+Alpha    2     60.00  2       1.50            A(fter rad
D: N+Gamma    3    100.00  3       5.00
E: Beta                    4       7.50              FILTER
F: Alpha                                           0 none
G: Gamma
H: BG
X: new Beta
```

Figure 36. Menu from which parameter values for disks are selected (Bortelot, 1990, figure 9).



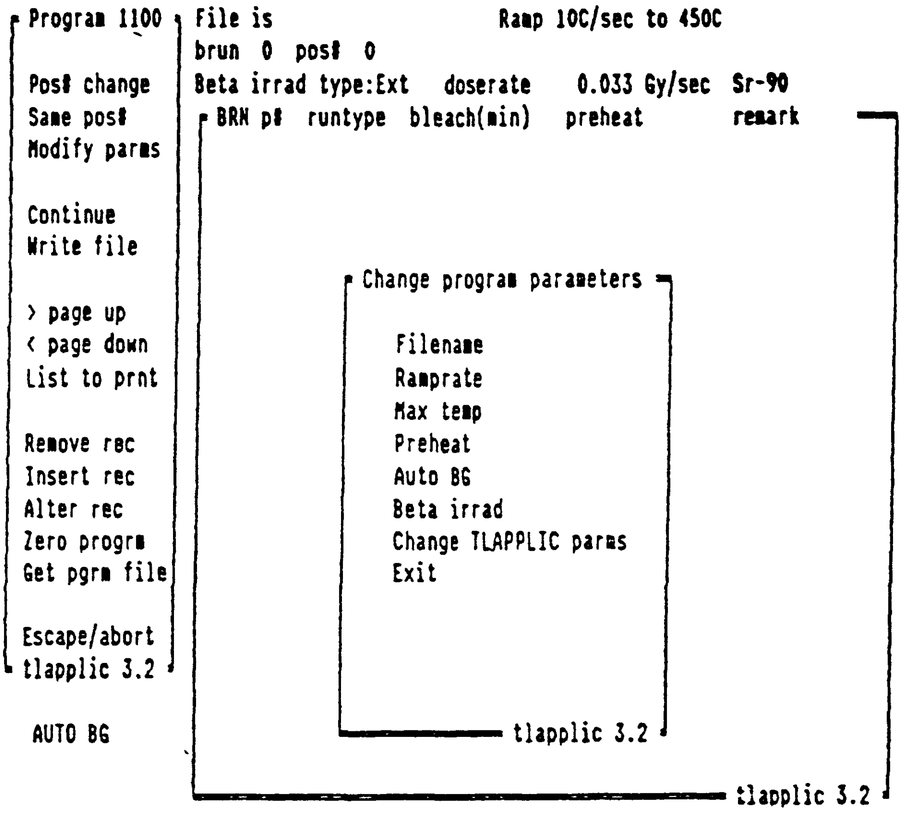
```
Program 1100   File is XXX.PRG           Ramp  5C/sec to 450C
               brun  5  pos#  4
Pos# change    Beta irrad type:Ext   doserate    0.033 Gy/sec  Sr-90
Same pos#       BRN p#  runtype  bleach(min)   preheat         remark
Modify parms     0  ON           B     0 none   0C  0s
                 1  Obackground  B     0 none   0C  0s
Continue         2  1N           B     0 none   0C  0s
Write file       3  2N           B     0 none   0C  0s
                 4  3N           B     0 none   0C  0s
> page up        5  4N           B     0 none   0C  0s
< page down
List to prnt

Remove rec
Insert rec
Alter rec
Zero progrm
Get pgrm file

Escape/abort
tlapplic 3.2
                                                          tlapplic 3.2
```

Figure 37. Batch program display screen after six runs have been entered (Bortelot, 1990, figure 10).



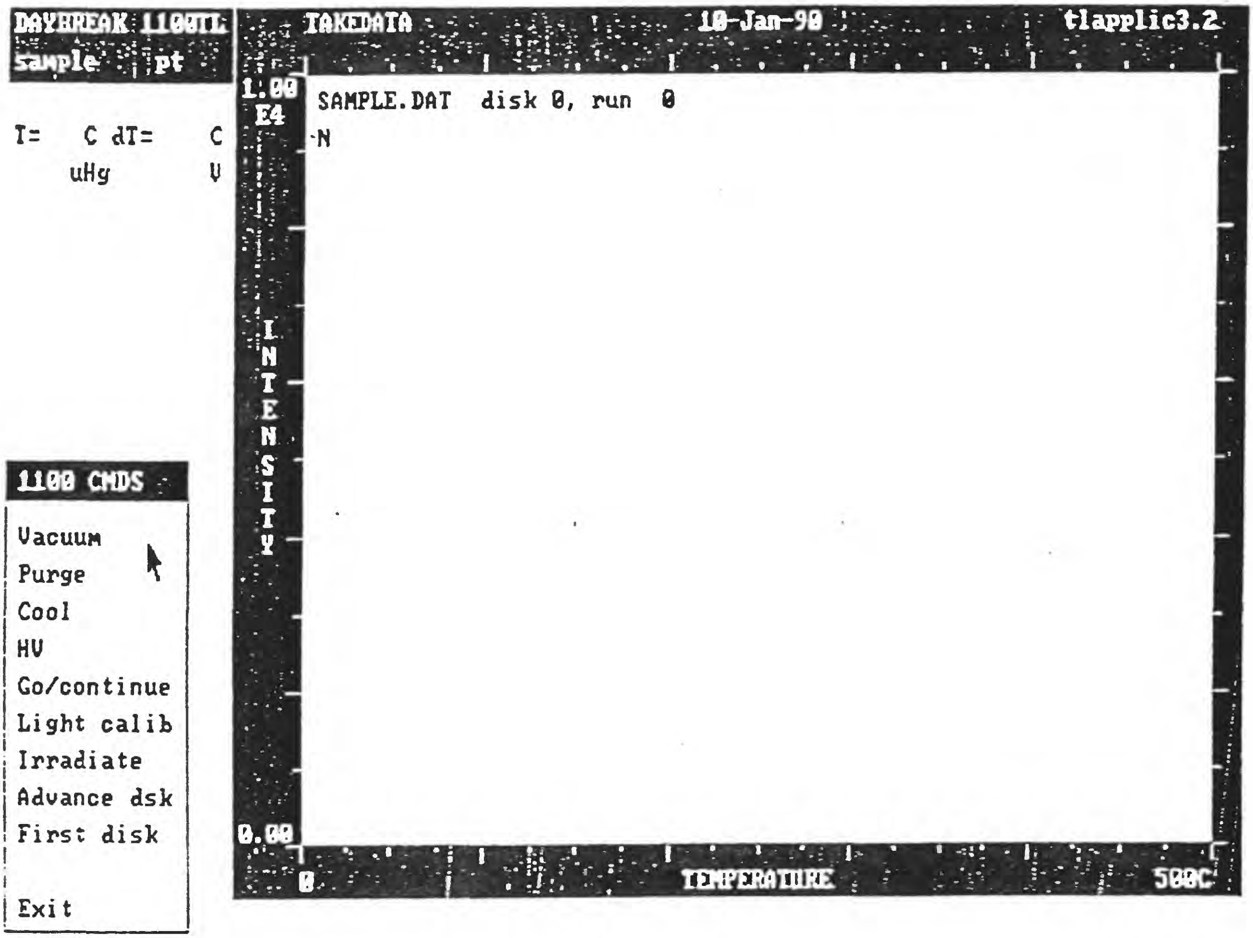
```
Program 1:00   File is                    Ramp 20C/sec to 500C
               run# 1 disk# 0
Disk# change   Beta irrad type:High  doserate   0.038 Gy/sec  Sr-90
Same disk#      Run d#  runtype  bleach(sec)  preheat        remark
Parameter ch    0  ON           B    0 none    0C  0s
                1  Obackground  B    0 none    0C  0s
Continue
Write file

> page up
< page down               Change disk number
List to prnt
                             I(ncrement disk# or <space>
Remove rec                   N(o change
Insert rec                   C(hange it
Alter rec
Zero progrm
                                              tlapplic 3.2
Escape/abort
tlapplic 3.2

AUTO BG

                                                        tlapplic 3.2
```

Figure 38. Menu to change next disk number (Bortelot, 1990, figure 11).



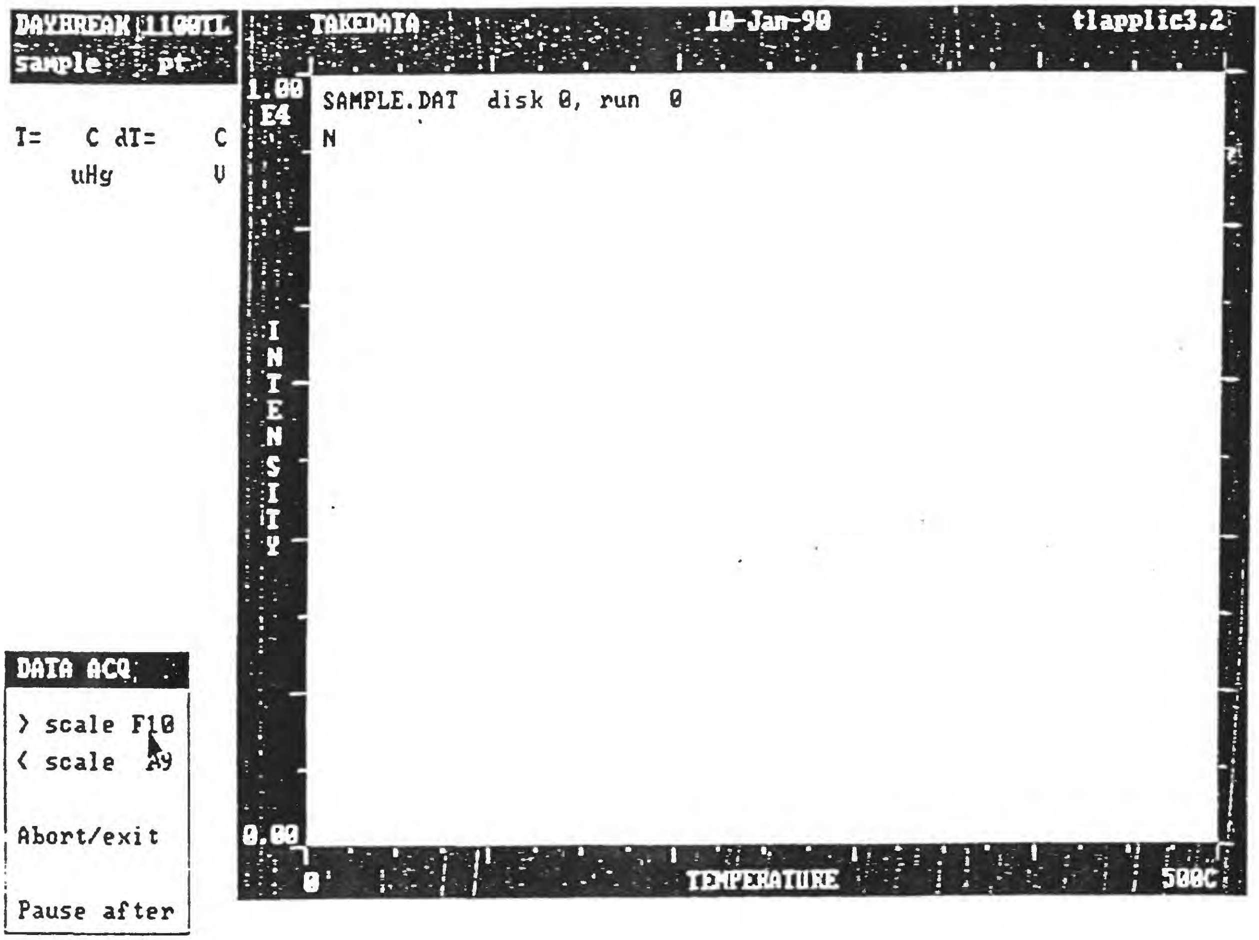
```
Program 1100   File is                 Ramp 10C/sec to 450C
               brun 0  pos# 0
Pos# change    Beta irrad type:Ext  doserate   0.033 Gy/sec  Sr-90
Same pos#       BRN p#  runtype  bleach(min)  preheat      remark
Modify params

Continue
Write file
                           Change program parameters
> page up
< page down                    Filename
List to prnt                   Ramprate
                               Max temp
Remove rec                     Preheat
Insert rec                     Auto BG
Alter rec                      Beta irrad
Zero progrm                    Change TLAPPLIC params
Get pgrm file                  Exit

Escape/abort
tlapplic 3.2

 AUTO BG                                  tlapplic 3.2

                                                         tlapplic 3.2
```

Figure 39. Menu to change batch program parameters (Bortelot, 1990, figure 12).



```
SAMPLE.DAT  disk 0, run  0
N
```

Figure 40. TAKEDATA screen with 1100 CMDS menu (Bortelot, 1990, figure 6).



SAMPLE.DAT disk 0, run 0

Figure 41. TAKEDATA screen with DATA ACQ menu (Bortelot, 1990, figure 7).