THERMOLUMINESCENCE OF QUARTZ IN SANDSTONE URANIUM DEPOSITS METHODS AND INITIAL TESTS

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INTRODUCTION

Mineral dosimeters such as quartz are particularly attractive for radiometric exploration because they are sensitive, have very good signal to background characteristics, and retain exposure information for geologically significant lenghts of time. Quartz is such a mineral, and the behavior of smoky quartz as a paleo-dosimeter has been noted by uranium prospectors for a long time. It was recently described by Saucier (1972). We have been investigating another dosimeter property of quartz--thermoluminescence--and evaluating its use as an exploration guide to sandstone uranium deposits. In this report we characterize the quartz thermoluminescent response as applied to uranium exploration, describe our sample handling, and present test results.

The thermoluminescence project at New Mexico Bureau of Mines and Mineral Resources was funded in part by the New Mexico Energy Resources Board grant #147. We have received generous cooperation from United Nuclear-Homestake Partners, Rocky Mountain Energy Company, and Exxon. The data presented in this report are derived almost exclusively from thermoluminescence measurements on quartz from the Westwater Canyon member of the Morrison formation in the Ambrosia Lake district, New Mexico. In this regard we are particularly indebted to the personnel of the United Nuclear-Homestake Partners, especially Bill Harrison, Chief Geologist, and his assistants, Jack Carter and Bernie Broadbent. We are very appreciative of the loan of a model 2000 TL analyzer by the Harshaw Chemical Company through Mr. Jack Owens.

THE PHENOMENON OF THERMOLUMINESCENCE

Thermoluminescence is an exceedingly complex phenomenon, and we will not consider the detailed theory in this publication. Those interested may wish to consult Thermoluminescence of Geological

Materials edited by D. J. McDougall (1968). Papers by Bonfiglioli

(p. 15), Levy (p. 25), and Braulich (p. 61) in this work treat the theory in some detail. Very briefly, when a mineral grain is exposed to ionizing radiation, mobile electrons and holes* are produced, and some of them become stored at structural defects or impurity sites called "traps." Trapped electrons can be released if sufficient energy is applied to the crystal, and when such electrons combine with a trapped hole, light is emitted. If the luminescence is due to the application of mechanical energy, it is called triboluminescence, and if it is due to the application of thermal energy it is called thermoluminescence, or TL.

In quartz, traps exist in several discrete populations and are drained at characteristic temperatures. It is convenient to refer to the traps by their drainage temperatures. In addition to drainage of traps by the external application of energy, spontaneous drainage occurs as a result of normal thermal vibration in the crystal. The probability of such drainage is less for higher temperature traps than for low, and one may characterize a mean lifetime of trapped electrons which varies

^{*} A hole is the absence of an electron.

from a few hours for 110°C traps in quartz to several tens of millions of years for traps in excess of 300°C.

Analytically, TL is measured by heating the quartz at a constant rate and recording the intensity of emitted light as a function of temperature. The intensity of light emitted during the measurement is proportional to the amount of trapped charge just prior to the measurement. This amount plus the trapped charge lost by spontaneous drainage is proportional to the ionizing radiation flux experienced by the crystal.

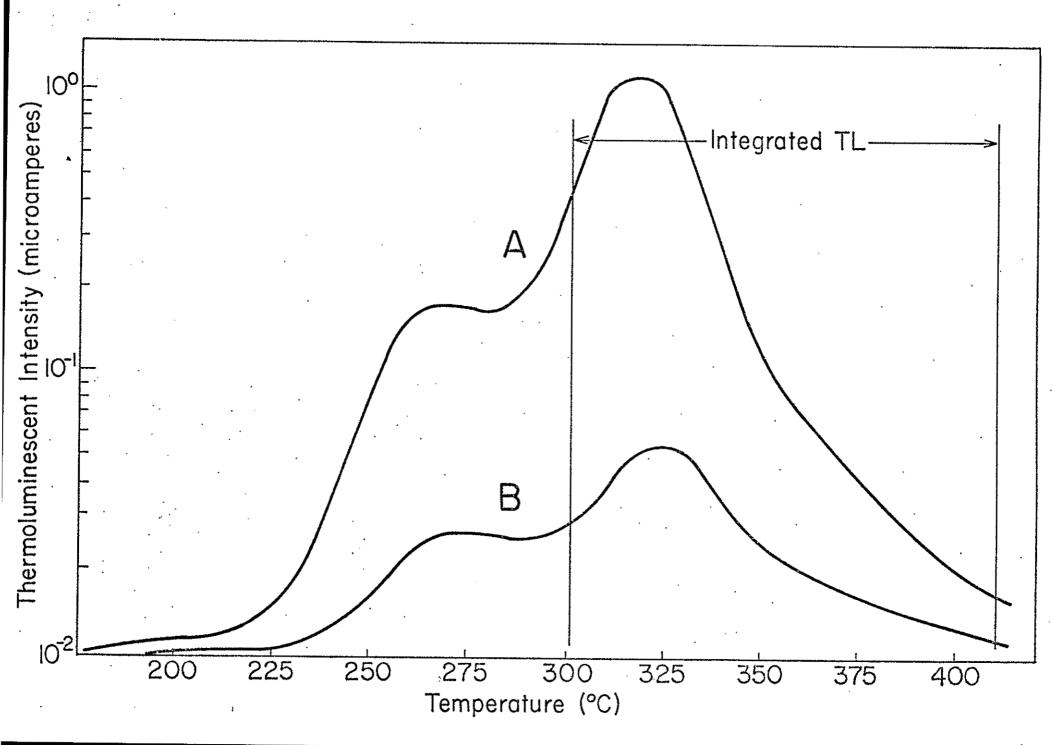
The TL output can be presented as a "glo:"curve, " which shows the distribution of luminosity as a function of temperature, or as the integrated luminosity in one or more temperature intervals. Typical glow curves of quartz samples are shown in figure 1.

Quartz has at least ten TL peaks between minus 110°C and 400°C (Medlin, 1968; Fleming, 1968), but for our purposes, only the 110°, 235°, 325°, and 375° peaks are significant. The two low temperature peaks at 110°C and 235°C retain electrons for a few hours to less than 100 years respectively.

The two high temperature peaks at 325°C and 375°C are important because they represent traps which retain electrons for geologically long periods of time (10⁸ years or more). The two peaks are poorly separated from each other on a glow curve, so their TL intensities are integrated in a single temperature interval. The TL due to drainage of high temperature traps is called natural TL and that due to drainage of temperature traps after artificial irradiation is called

Figure 1:

Representative glow curves of quartz thermoluminescence from the Highland Mine (EXXON), Powder River basin, Wyo. Curve A is from a sample of low grade ore; curve B is from a sample of oxydized ground 600 feet away from ore. The 375°C peak is unresolved in these samples. The temperature range over which integrated TL is measured is shown.



artificial TL. In this report we will be concerned primarily with natural TL.

In the geological environment, quartz collects a radiation dose for an extremely long period of time and records gamma-radiation from sources considerably more remote than can be detected by industrial instrumentation operating routinely. In addition, the radiation record is stored in the quartz crystal lattice for geologically significant lenghts of time (e.g. 10⁸ years). A consequence of these two properties is that a single anomolous TL determination in the absence of an anomalous radionuclide concentration in the sample is inherently ambiguous: one cannot decide from it whether the TL response is due to an ancient high radiation flux now gone or due to a concealed radiation source nearby.

To resolve this ambiguity, several TL measurements must be made at appropriate intervals along a traverse. If anomalous TL variation shows a positive correlation with radionuclide variation along the traverse, we call the variation a type I anomaly. If there is no correlation with radionuclide concentration we call it a type II anomaly. Of course, if a type II anomaly is followed into ore it becomes a type I anomaly close to and within the orebody.

Both type I and type II TL anomalies are of value in uranium exploration. The type I anomaly is applicable in terrain where uranium is fixed, as in a reducing environment. The type II anomaly is applicable where uranium has been mobile, as in an oxydizing environment.

The TL project at New Mexico Bureau of Mines has concentrated

on evaluating and characterizing the type II anomaly. This report deals with the sample preparation techniques, instrumental method, and initial test results. For an exhaustive treatment of the theory, method, and experimental results, the reader is referred to Hayslip (1976).

SAMPLE PREPARATION

Samples were collected from the Westwater Canyon Sandstone member of the Morrison formation in the underground workings of the Section 23 and Section 25 mines in the Ambrosia Lake district, New Mexico. See Fig. 2. This sandstone is an arkose which varies from highly friable to indurated by carbonate cement. The concentration of quartz in these rocks is on the order of 50 percent; however, the sample preparation procedure yields about one-half weight percent of the rock suitable for TL determination. Samples weighing about 100 grams were collected in cloth bags.

Protection

The first precaution we observed was to protect the samples from ultra-violet radiation. A number of studies have shown that the absorbtion of UV radiation enhances some parts of the TL spectrum and diminishes others. Following Aitken and Flemming (1972) we stored samples in the dark and conducted sample preparation under red light. We made no tests on the necessity of this procedure, and we may have been over cautious, for the effects of UV radiation are most noticeable at low TL levels, and the magnitude of the TL anomalies we observed are very large.

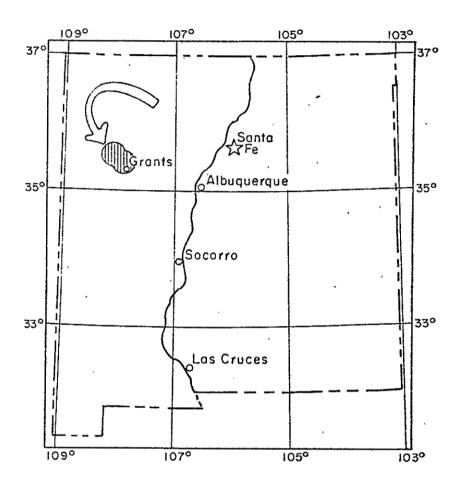


Figure 2: Location of the Ambrosia Lake Mining District, New Mexico

Sample preparation in the lab is divided into three parts:

1) disaggregation, 2) size separation, and 3) cleaning.

<u>Disaggregation</u>: We avoided grinding of samples in order to eliminate the introduction of spurious TL drainage due to triboluminescence. The friable nature of much of the material eased sample preparation in this respect considerably.

About 75 grams of sandstone were presoaked in about 30 ml of 10% HCI solution for 8 to 24 hours. This procedure softens clays and dissolves a small amount of carbonate and iron oxide.

After the presoak, samples were rinsed in clean water and disaggregated in 10% HCI by eight 10 minute treatments in an ultra-sonic cleaner. After each treatment, the sample was rinsed with clean water and placed in fresh 10% HCI solution. It was thought that the input of mechanical energy due to the ultrasonic treatment might drain some of the TL so the effect was tested and found unimportant. After the final ultrasonic treatment, the samples were washed in clean water followed by acetone to hasten drying.

Sizing: After disaggregation and drying, the sample is sieved to obtain a uniform size fraction. It was found that the size fraction from 106 microns to 177 microns gave a clean magnetic separation and consistent TL response. In this size range, the contribution of the distribution to the total TL is negligible.

Although it was not necessary to grind the Westwater canyon samples to obtain an appropriate size fraction, a grinding test was conducted. After disaggregation and removal of the 104-177 micron

fraction by seiving, the coarse and fine fractions were mixed together and divided into two equal aliquots of approximately 20 grams each.

One aliquot was ground by hand for two minutes in a porcelain mortar at room temperature. The other aliquot was ground in the same mortar, but under liquid nitrogen for two minutes.

Table 1

	Unground	Ground at room temp.	Ground in lig. N ₂
Mean TL (nanocoulombs)	1544	1272	1548
Standard deviation	87	58	106
No. of replications	10	8	8

Following the grinding, samples were seived to obtain a 104-177 micron sample from each aliquot. The results are given in Table 1 above.

Further tests should be conducted, but degradation of TL by grinding at room temperature is clearly indicated, and it seems likely that grinding at low temperatures may solve the problem.

Cleaning: After the sample has been sized, it is passed through a Franz isodynamic separator to concentrate a pure fraction of clean quartz. The separator is operated at an inclination of 10 degrees and a cross slope of minus \frac{1}{2} degree. The separator magnet is run at its maximum setting of 1.8 amps. The sample is passed through the separator one time at a very slow rate; it takes about an hour to process 5 grams of sample. The final yield is a 250 to 500 mg separate containing about 90 percent quartz.

MEASUREMENT OF QUARTZ THERMOLUMINESCENCE

Measurements of quartz thermoluminescence were obtained with a model 2000 thermoluminescence analyser manufactured by Harshaw Chemical Company. The unit is illustrated in figures 3 and 4. The model 2000 is an off-the-shelf instrument and we have made no modifications to it. The unit consists of two instruments -- the TL detector and the picoameter. They are operated simultaneously from a 115 volt line. At this writing, total cost for equipping a TL lab exclusive of sample preparation facilities is of the order of \$7,000.

The detector geometry is shown in figure 4. It consists of a platinum planchet which serves as a resistance heater and sample heater, an optical train, and an electronically cooled photomultiplier tube. A vibrating volumetric sample dispenser is attached which charges a shallow square depression in the planchet. Provision is made for drifting dry nitrogen over the sample to control the atmosphere in which TL measurement is made.

The output of the photomultiplier tube is scaled on a sensitive auto-ranging picoameter and integrated between adjustable temperature limits. TL intensity is digitally expressed in nanocoulombs (nc). Output jacks permit interfacing an X-Y recorder and a printer.

The quartz sample is introduced into the heating planchet by means of the volumetric dispenser supplied with the apparatus. The mean weight dispensed for the grain size 106 microns to 177 microns is 18.0 mg with a standard deviation of 0.3 mg for 15 replications.

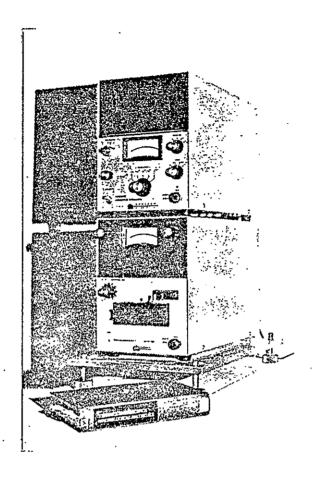


Figure 3: Harshaw Model 2000 TL Analyser mounted on leveling stand.

The high elasticity of quartz requires that grains be constrained from bouncing out of the planchet during loading. A square tubular loading chute whose cross-section is congruent with the planchet depression was used to eliminate sample loss due to this behavior (see figure 5).

The amount of light emitted from the sample is a function of the amount of sample and its geometry, so pains must be taken to assure the reproducibility of these instrumental conditions. Uniform grain size and uniform distribution in the depression of the planchet are as important as uniform sample weight. We found that placing the apparatus on a platform whose inclination could be adjusted with leveling screws aided considerably in obtaining a uniform distribution of quartz grains in the planchet depression. Vibrations of the dispenser are transmitted to the planchet by means of a small wooden stick in order to further assure that the quartz grains are distributed as a uniform layer in the planchet depression.

After the sample has been loaded into the planchet depression, it is moved into the optical train, and the heating chamber is flooded with dry nitrogen at the rate of five liters/min to eliminate spurious TL response due to the presence of oxygen as recommended by Aitken and Flemming (1972). After two minutes, the atmosphere in the heating chamber is essentially pure nitrogen; heating is begun and the thermoluminescent response of the sample is recorded. For the purposes of this study, natural TL response was integrated between 300° and 410° C.

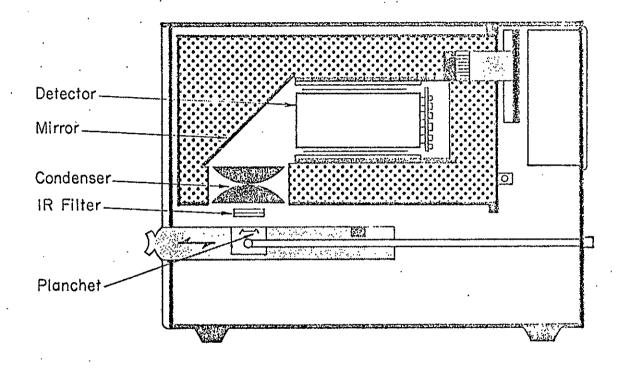


Figure 4: Schematic diagram of the Harshaw Model 2000 analyser.

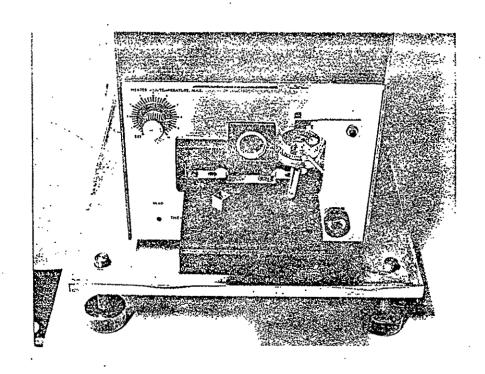


Figure 5: Detail of loading chute, planchet containing a sample, and the sample dispenser. The infra red rejecting filter is mounted on a hinge above the planchet.

Under the laboratory conditions at the New Mexico Bureau of Mines, it was found that replicate determinations of TL must be regularly spaced in order to obtain reproducible results. Generally the first determination is rejected, and subsequent determinations follow at two minute intervals.

The TL heater was calibrated by mean of temperature sensitive paint called TEMPILAQ which is supplied by Omega Engineering, Inc.

This product is claimed to be accurate to plus or minus 0.5°C.

The heating rate potentiometer was calibrated by means of a stop watch and visual observation of the thermocouple galvanometer over the interval from 200° to 400° C. The heating rate for glow curve plotting is $4.0 \pm .1^{\circ}$ C/sec and the rate for determination of integrated TL is $12.0 \pm .1^{\circ}$ C/sec. The errors given here are one standard deviation.

The stability of the temperature limits of TL integration were determined by removing the IR rejecting filter and recording the black-body emission of the furnace between 300° and 410°C at five minute intervals. Five replications gave a coefficient of variation of 0.012.

The stability of the Harshaw instrumentation is very good and results have been found to be highly reproducible over the several months duration of our research. No attempt has been made to calibrate the output of the picoameter, so the magnitude of our measurements of thermoluminescence intensities are not directly comparable to those of other workers. However, all our results are comparable to each other at a high level of precision.

PRELIMINARY TEST RESULTS

Introduction

Preliminary testing of thermoluminescence effects in quartz from sandstone uranium deposits was conducted on material from the Powder River basin, Wyoming and from the Ambrosia Lake Mining district, McKinley County, NM. The results presented in this report are based on material collected from the Section 23 and Section 25 mines of United Nuclear-Homestake Partners in the Ambrosia Lake district. These mines are located in sections 23 and 25, T. 14 N., R. 10 W. See Figure 6 for traverse locations. In the section 23 Mine, all samples in oxydized ground were collected on the 726 level which is in the lower third of the Westwater Sandstone member of the Morrison Formation. Samples in reduced ground were collected on the 650 level which is the middle of the Westwater Sandstone member. Replication

Results reported elsewhere (Hayslip and Renault, 1976) have shown that the range of integrated quartz thermoluminescent response near uranium deposits is about 5500 nanocoulombs at Ambrosia Lake, New Mexico. Table 2 shows means and coefficients of variations of TL integrated from $300\,^{\rm O}$ C to $410\,^{\rm O}$ C for seven representative samples from the Westwater Canyon sandstone exposed in the Section 23 Mine in the Ambrosia Lake Mining district. Replicate determinations of N = 3, N = 5, and N = 10 are given for each sample. These data show

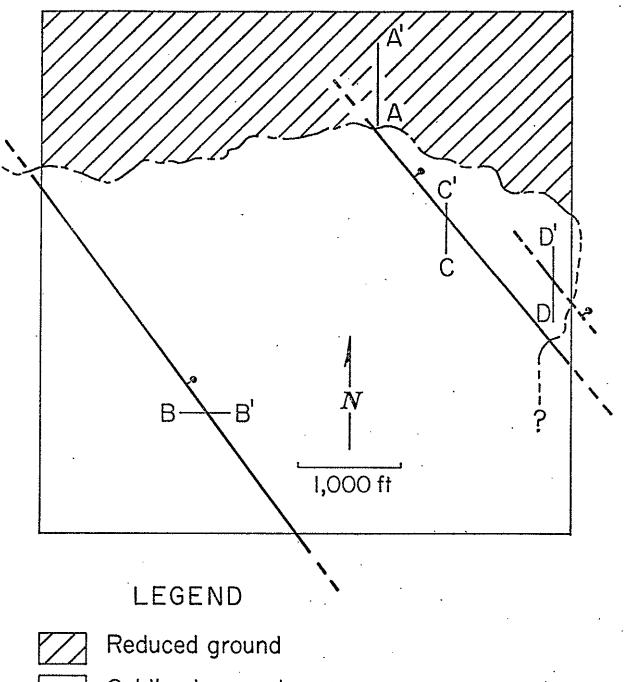
that even with ten replications, there are occasionally samples whose coefficients of variation exceed 0.10. However, the mean coefficient of variation plus its standard deviation for ten replications is only 0.0947. For replications of N = 5 and N = 3, the mean coefficients of variation plus their standard deviations are 0.106 and 0.115 respectively.

For exploration purposes where relative values of TL are more important than absolute values in establishing the sense of anomaly, three replications are probably adequate. As an illustration of this, notice that for ten replications the mean integrated TL of samples No. 7 and No. 116 differ by only 0. 14 x 10³ nanocoulombs. Using the best means and standard deviations of these two samples, it can be shown that if three replications are made on each sample, the probability of the mean TL from No. 7 being greater than that of No. 116 is only 0.01.

Representative sampling

In order to evaluate the short distance variability of TL, a suite of 16 samples of Westwater Canyon sandstone was collected on a square grid pattern at 2 ft. intervals on the 726 level of the Section 23 mine. The distribution of integrated 300° - 410° C TL is shown in Table 3 along with equivalent uranium concentrations for each sample. The distribution of values appears to be random by visual inspection. The mean and coefficient of variation of the TL for the 16 samples are 1.23×10^{3} nanocoulombs and 0.079; note that the coefficient of variation is of the same order as that found for several replications of the same sample.

Figure 6: Location map of underground traverses within the Westwater sandstone in Section 23, T.14 N., R.10 W.



Oxidized ground

-- Redox boundary (dashed where inferred)

High-angle normal faults showing downthrown side (dashed where inferred)

TABLE 2: Mean integrated TL and coefficients of variation for various replications over the high temperature peaks of quartz from the West Water Sandstone.

Sample No	N = 3		N = 5		N = 10	
	\bar{x}_{TL}^*	$\mathtt{c}_{\mathtt{TL}}$	X _{TL}	C _{TL}	\bar{x}	$c_{_{\mathrm{TL}}}$
37	.756	.0477	.804	.110	.817	.102
7	1. 12	.0402	1.13	.0691	1. 12	.0740
116	1.19	.0513	1.22	.0545	1.26	.0505
89	1.83	.0852	1.85	.0670	1.80	.0561
312 B	2.34	.0500	2.33	.0380	2.33	.0297
590	2.66	.117	2.58	.0942	2.69	.0918
302 A	3.69	. 103	3.55	.0943	3.49	.0822
Mean C		.0707		.0753		.0693
Std. dev. of C .0308		,	.0253		.0254	

^{*} \overline{X}_{TL} is nanocoulombs $\times 10^3$

It is important to evaluate the effect of local radionuclide concentrations on the measured thermoluminescence. Equivalent uranium (eU) concentrations obtained by gamma-ray spectrometry are shown in Table 3 along with the TL measurements. Their mean and coefficient of variation are 5.08 ppm and 0.95. One particularly high eU concentration of 24.17 ppm was determined for the sample in column 1, row 3, but this sample does not have a particularly high TL measurement. Likewise, the sample in row 1, column 4 has the highest TL value, but has a somewhat low eU concentration. In fact there is no conspicuous correlation between TL and eU, and the calculated correlation coefficient is only 0.04, whether the two extreme values are included or not.

TABLE 3
Grid Sampling Test for Short Range Variability

1.27	1.18	1.20	1.57
(4.2)	(4.3)	(3.2)	(4.0)
1.20	1,22	1.20	1.15
(2.1)	(4.3)	(4.2)	(4.0)
1.25	1.22	1.17	1.20
(24.)	(5.2)	(4.4)	(4.1)
1 20	. 10		
1.23	1.19	1, 31	1.18
(4 .9)	(3,6)	(4.3)	(5.0)

Smaller values are nanocoulombs (X10³)
Values in parentheses are equivalent uranium concentrations in ppm. Means and coefficients of variation of TL and eU are 1.23, .079 and 5.37, .95 respectively.

THERMOLUMINESCENCE ANOMALIES IN THE SECTION 23 MINE

In the Ambrosia Lake district and in the Section 23 Mine in particular, there are two main classes of ore which are called, in nongenetic terms, trend ore and stack ore. These ores occur throughout the Westwater Canyon sandstone member of the Morrison formation in Section 23 T. 14 N., R. 10 W. (Santos, 1963), but trend ore predominates in the southern part of the section and stack ore in the northern part.

Trend ore is thought to be primary in origin, that is, it represents the earliest concentrated uranium precipitation after deposition of the host rocks. It is coextensive with organic material and occurs in linear to tabular bodies trending approximately N 70°W. Stack ore is thought to be secondary in origin, that is, it represents the redistribution of primary (trend) ore by groundwater movement down the hydrologic gradient. Primary ore at Ambrosia Lake is of the order of 100 m.y. old redistributed (stack) ore is of the order of 10 m.y. old (Dooley, et al., 1966). Primary ore is simply cut and displaced by Larimide and post-Larimide faults; redistributed ore is controlled in position and geometry by Larimide and post-Larimide faults (Squyes, 1970). Primary ore presently occurs in both oxidized and reduced ground; redistributed ore occurs at the boundary between oxidized ground and reduced ground.

As mentioned earlier, the rationale of this study is that fixed as well as migrating anomalous uranium concentrations generate

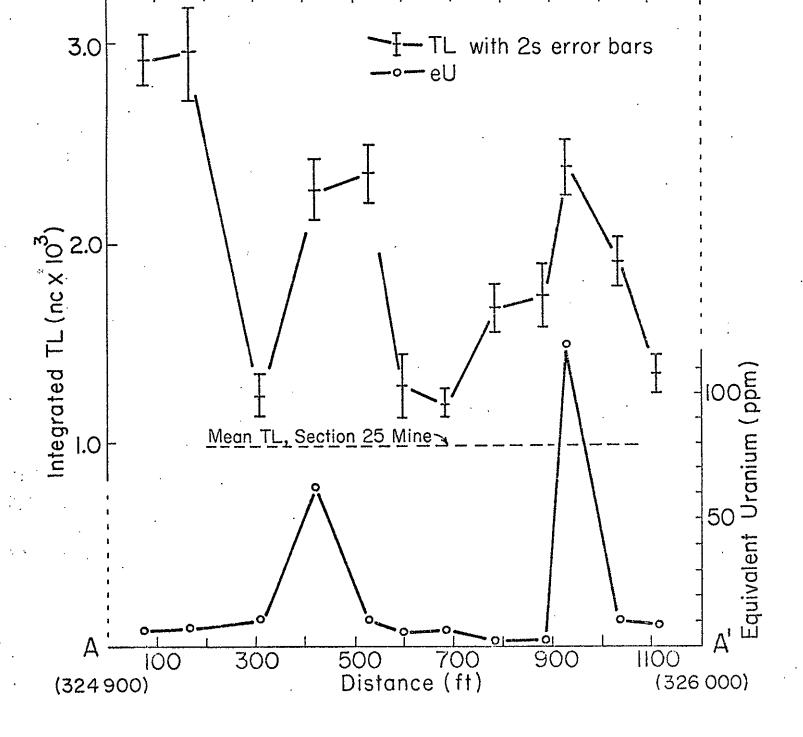
anomalous trapped charge concentrations in quartz of the host sandstone. The anomalous trapped charge concentration is revealed by thermoluminescence measurements on the quartz. Two types of TL traverses in the Section 23 mine illustrate type I and type II anomalies associated with redistributed ore: (1) one N-S traverse approximately normal to the redox interface ore in reduced ground and (2) three traverses across faults in oxidized ground which project into redistributed ore. See figure 6.

The type I anomaly normal to the redox interface is shown in figure 7. The traverse was made in the 650 level 3400 ft east of the section 22-section 23 boundary and extends due north from just beyond the redox interface for 1100 feet. Samples were collected in reduced ground at approximately 100 ft. intervals. The significant feature of this traverse is the dependence of TL magnitude on equivalent uranium concentration. As is expected, the long range recording ability of the quartz TL phenomena reveals broader anomalies than are shown by gamma-ray spectrometry. The four lowest TL determinations are significantly above the mean TL value* for reduced ground in the Section 25 mine. This may indicate that there has been a small amount of mobilization of radionuclides beyond the redox interface.

As part of our TL study, we made a series of traverses across post-Laramide faults in the Section 23 Mine. The locations of the traverses are shown in figure 6. Typical type II TL anomalies observed

^{*} This is the mean of the six lowest TL measurements at distances greater than 100 ft. from primary ore. $\bar{x} = p.98 \times 10^3$ nc, $s = 0.05 \times 10^3$ nc.

Figure 7: Type I TL anomaly in reduced ground. Traverse A-A' in Fig. 6. The mean TL in barren reduced ground of the section 25 mine is shown for comparison.

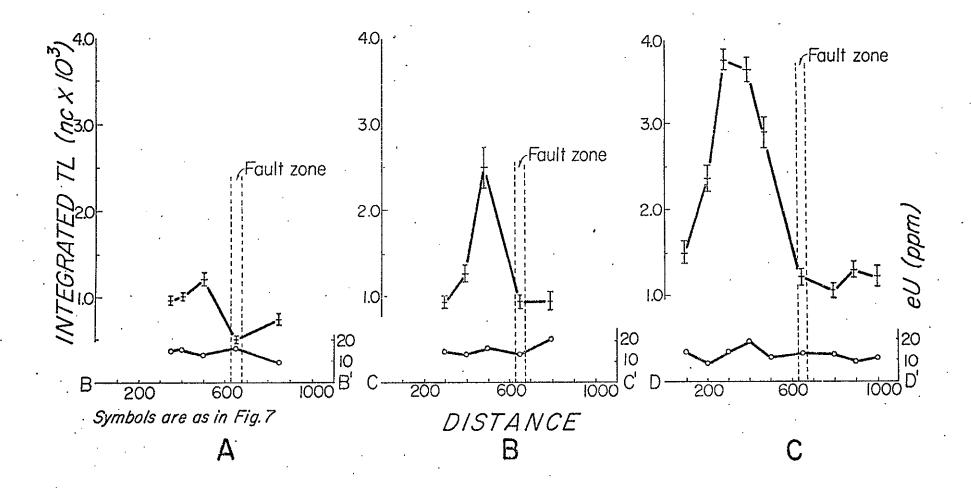


on such traverses are shown in figures 8A, B, and C. Traverses were made in oxydized ground with a sampling interval of 100 ft beyond the faults and 10 feet within the faults. In these figures the TL values plotted within the fault zones are the averages of the several samples collected within the respective fault zones.

Three important features seen in figures 8A, B, and C are the independence of TL and eU, the restriction of the TL anomalies to the SW side of the faults, and the apparent increase in the amplitude of the TL anomalies as stack ore is approached. The significance of the independence of TL and eU has been discussed above—it defines the type II anomaly in oxydized ground. The restriction of TL anomalies to the SW side of faults is consistent with the observations of Gould, et al. (1968), Squires (1970), and others that young faulting in the Ambrosia Lake district has controlled the spacial redistribution of uranium. It is beyond the scope of this report to analyse these observations in detail, but if groundwater movement at the time of redistribution was from the southwest, the implication is very strong that the faults deflected its movement, and the increase in the amplitude of the TL anomalies with approach to stack ore further implies that the faults guided the uranium redistribution.

It should be noted in figures 8A, B, and C that the maxima of the TL anomalies for strike distances of 3000 ft, 800 ft, and 250 ft stack ore are 1.25×10^3 nc, 2.5×10^3 nc, and 3.8×10^3 nc respectively. It will be appreciated that the persistence of the type II TL anomaly for distances of the order of 3000 feet from redistributed ore is of considerable importance in uranium exploration.

- Figure 8 A: Type II TL anomaly associated with a fault in oxydized ground. Traverse B-B', 3000 ft. from redistributed uranium ore.
- Figure 8 B: Type II TL anomaly associated with a fault in oxydized ground. Traverse C-C', 800 ft. from redistributed uranium ore.
- Figure 8 C: Type II anomaly associated with a fault in oxydized ground. Traverse D-D', 250 ft. from redistributed uranium ore.



SUMMARY AND CONCLUSIONS

Quartz thermoluminescence associated with sandstone uranium deposits has been evaluated from the standpoints of sample preparation, instrumentation, reproducibility, and relationship to redistributed uranium ore. Sample preparation is a four step process involving (1) protection of samples from UV radiation, (2) disaggregation of compact samples to separate quartz from matrix material such as carbonate and clays, (3) sizing the disaggregated sample to obtain a fraction coarse enough to minimize complications due to alpha-radiation in the sample and uniform enough to yield consistent thermoluminescence, and (4) cleaning with a magnetic separator to assure that the thermoluminescent phenomena observed are due to quartz and not complicated by the presence of other phases such as feldspar and iron oxides.

The instrumentation used for this project was the Harshaw Model 2000 thermoluminescence analyzer routinely used in the dosimetry industry. It is an "off the shelf" item and required no significant modification for application to this study.

Tests of reproducibility showed that the precision of TL measurement is substantially better than 10 percent for measurements reported here when ten replicate determinations are made on each sample.

Precision is of the order of 10 percent for three replications, but for samples whose TL output differs by only 0.1 x 10³ nc, the probability that their relative magnitudes be reversed is only 0.01. It was found that over an area of 64 square feet, 16 samples showed no more TL variation than replications of a single sample. No correlation with

equivalent uranium was observed in this test.

Thermoluminescence traverses in the Section 23 Mine, Ambrosia Lake district, McKinley Co., N.M. illustrate type I and type II TL anomalies. Type I anomalies occur in reduced ground and TL variation correlates with equivalent uranium concentration. Type II anomalies occur in oxydized ground and the TL variation shows no correlation with eU. Type II anomalies were observed to occur on the SW side of NW-SE trending post-Larimide faults. These anomalies increase in amplitude with the approach to stack ore.

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