

1 **Thermoluminescence and Apollo 17 ANGSA lunar samples: NASA's fifty-year experiment**
2 **and prospecting for cold traps.**

3 Derek W. G. Sears¹, Alexander Sehlke¹, Harrison H. Schmitt² and the ANGSA Science Team

4 ¹NASA Ames Research Center/ Bay Area Environmental Research Institute
5 Moffett Field, California 95035, USA.

6 ²Department of Engineering Physics, University of Wisconsin-Madison, P.O. Box 90730,
7 Albuquerque, NM 87199-0730, USA

8
9 Corresponding author: Derek W. G. Sears

10 Email: dsears@uark.edu

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13
14 **Abstract**

15 By placing Apollo 17 regolith samples in a freezer, and storing an equivalent set at room
16 temperature, NASA effectively performed a fifty-year experiment in the kinetics of natural
17 thermoluminescence (TL) of the lunar regolith. We have performed a detailed analysis of the TL
18 characteristics of four regolith samples; a sunlit sample near the landing site (70180), a sample 3
19 m deep near the landing site (70001), a sample partially shaded by a boulder (72320), and a
20 sample completely shaded by a boulder (76240).

21 We find evidence for a total of eight discrete TL peaks, five apparent in curves for samples in the
22 natural state, seven in samples irradiated in the laboratory at room temperature. For each peak
23 we suggest values for peak temperatures and the kinetic parameters E (activation energy, i.e.
24 “trap depth”, eV) and s (Arrhenius factor, s⁻¹). The lowest natural TL peak in the continuously
25 shaded sample 76240 dropped in intensity by 60±10% (1976 vs. present room temperature
26 samples) and 43±8% (freezer vs room temperature samples) over the 50-year storage period,
27 while the other samples showed no change. These results are consistent with the E and s
28 parameters we determined.

29 The large number of peaks, and the appearance of additional peaks after irradiation at room
30 temperature, and literature data, suggest that glow curve peaks are present in lunar regolith at
31 ~100 K and their intensity can be used to determine storage times at these temperatures. Thus a
32 TL instrument on the Moon could be used to prospect for a micro-cold traps capable of
33 deposition, build-up and storage of volatiles.

34

35 Plain Language Summary

36 Rocks, like the "soil" (regolith) from the Apollo 17 landing site, glow when heated in the dark.
37 This glow, known as thermoluminescence (TL), is caused by previous exposure to radiation but
38 it can fade depending on ambient temperatures. We therefore have a method for studying
39 radiation and thermal history of these rocks, but we need to learn the precise details of this
40 process. Nearly fifty years ago NASA placed some Apollo 17 regolith (1) in a metal cabinet at
41 room temperature and (2) in a freezer. We found that freezer samples did not lose any TL, but
42 the cabinet samples showed considerable fading relative to the freezer samples and relative to
43 samples measured nearly fifty years ago. Combined with a detailed computer analysis of the
44 data these results enable us to understand the relationships between TL, time, and environmental
45 conditions. Most importantly, this method will enable us to search for tiny locations, called cold
46 traps, in the polar regions of the Moon where water and other volatiles may have accumulated.
47 This information is important for understanding the history of the Moon and it will support
48 exploration efforts which need water and volatiles.

49

1. Introduction

50 Thermoluminescence (TL) is the light emitted by a sample as it is heated (Boyle, 1664; Herschel,
51 1899; Sears et al., 2013; also see the Supplement). A plot of light emitted vs. heating
52 temperature is referred to as the "glow curve". Within the glow curve the luminescence is
53 emitted as a series of peaks, each reflecting a different defect or impurity in the crystal structure
54 where electrons can be "trapped". The level of luminescence naturally present in a given peak
55 depends on previous exposure to radiation, which increases TL levels, or to heat, which
56 decreases TL levels. Each peak has an activation energy E (eV, usually referred to as "trap
57 depth") and an Arrhenius factor s (s^{-1} , essentially a rate constant) which describe the kinetics of
58 light production. Thermoluminescence has practical applications in personnel dosimetry, pottery
59 dating, and authenticity dating of artifacts (Aitken, 1985; Horowitz, 2021). When Apollo
60 samples were returned from the Moon there was considerable interest in their TL properties (e.g.,
61 Hoyt et al., 1971; Durrani et al., 1972; Garlick and Robinson, 1972, Dalrymple and Doel, 1970;
62 Blair et al., 1972a,b). Most publications dealt with TL as a means of investigating the thermal
63 and radiation environment on and in the regolith (e.g., Hoyt et al., 1971; Durrani et al., 1976),
64 while some considered TL as a possible explanation for transient lunar phenomena (Geake and

65 Mills, 1977; Geake et al., 1977). Batchelor et al. (1997) have used the induced TL (as opposed
66 to natural TL) to deduce information on the petrologic and mineralogic history of lunar samples.
67 Induced TL is the level of luminescence displayed by the sample after its natural TL had been
68 removed by heating to 500 °C. Inducing a TL signal also reveals the existence of shallow
69 electron traps that were empty in the natural sample.

70 This paper concerns regolith samples from the Apollo 17 landing site in the Taurus-Littrow
71 valley (Schmitt, 1973; Wolfe et al., 1981). Harrison Schmitt and Gene Cernan collected ≥ 120 kg
72 samples from the Taurus-Littrow valley in December 1972. In three EVAs they traversed about
73 30 km in order to sample ancient pre-and post-Imbrium highlands, basalts, and ejecta from the
74 various impacts and pyroclastics deposits on the valley floor (Fig. S1). The present study
75 concerns regolith (1) from the landing site, (2) from 3 m depth near the landing site, and from the
76 foothills of (3) the North Massif and (4) the South Massif. The regolith consists of a mixture of
77 subfloor basalt regolith, volcanic ash, regolith from of the nearby massifs and unconsolidated
78 surficial material generated mainly from impact (Wolfe et al., 1981). The foothills sites were
79 notable for the number of boulders that had rolled down the Massifs and some of our samples
80 had been completely or partially, continuously shaded by the boulders for periods of about 20
81 Myr (North Massif) (Cozaz, et al., 1974) and 52 Myr (South Massif) (Leich, et al., 1975).
82 Schmitt et al (2017) reported a synthesis of data related to the Apollo 17, 3 m deep drill core and
83 found that it consists of 10 regolith ejecta zones laid down over about 3.3 billion years.

84 The most recent mass-wasting event originating from the slope of the South Massif is the
85 “young” light mantle of avalanche-derived material, originally thought to have been deposited as
86 a result of the impact of ejecta from Tycho Crater some ~2350 km to the southwest (Fig. S1;
87 Arvidson et al., 1976; Drozd et al, 1977; Lucchitta, 1977). An ejecta ray from Tycho crosses
88 Taurus-Littrow and some authors have argued that craters in the Crater Cluster, several
89 kilometers east of the old and young light mantles, have similar ages to the Tycho impact.
90 Alternatively, many large mass wasting events from the South Massif at different times may
91 have deposited seven mass-wasting deposits, including the most recent, “young light mantle”
92 investigated by Apollo 17 astronauts. The existence of the Lee-Lincoln thrust fault in the same
93 part of the valley as these mass-wasting events suggests that repeated seismic activity along this
94 fault may have triggered these events. Furthermore, recent work on the ages of 400-800 m

95 diameter craters in the Crater Cluster and their regolith ejecta (sampled by the deep drill core),
96 indicate that the Cluster is comprised of at least five different impact events, including four
97 elliptical, apparently simultaneous impacts of that may be from a cometary aggregate. These new
98 findings indicate that Tycho ejecta only could be responsible for only one of the mass-wasting
99 events. Arguing against even that possibility is the youngest cosmic ray exposure age for ejecta
100 from the Crater Cluster is 360 Myr (Eberhardt, et al., 1974) versus an age of ~52 Myr (Leich, et
101 al., 1975) for the youngest mass-wasting event, the young light mantle.

102 Durrani et al. (1976) used TL measurements to discuss the thermal history of these samples
103 pointing out that samples collected in the shadow of a large boulder had a stronger TL signal
104 than partially shaded samples which in turn had slightly stronger signals than samples collected
105 in direct sunlight. These authors described in some detail the theoretical underpinning of the
106 measurements and how an equilibrium temperature (which they called “storage temperature”)
107 could be derived. Since the discovery of water on the Moon in permanently shadowed craters
108 (Nozette et al., 1996; 2001), and the prediction of water-bearing micro-cold traps (Hayne et al.,
109 2021), we have pointed out that TL could be used to prospect for locations suitable for the
110 retention of water ice and other volatiles (Sehlke and Sears, 2022). Schmitt (2023) also stressed
111 the value of thermoluminescence measurements in understanding the thermal history of the lunar
112 regolith at high latitudes.

113 Durrani (1972) pointed out that it is possible that samples associated with particularly low
114 temperatures or recent radiation exposure could have TL peaks that are unstable at room
115 temperature and he therefore advocated storing returned lunar samples in a freezer. In the run-up
116 to the return of humans to the Moon by Artemis, NASA made available samples of Apollo 17
117 regolith that had been stored in a freezer for nearly fifty years (1973-2022). They also released
118 the equivalent room temperature samples. This provided a unique opportunity to characterize the
119 natural TL of lunar samples and understand the kinetics of natural TL build-up and decay. This
120 is essential if TL is to be used for science and exploration, particularly in the case of water and
121 volatile prospecting.

122

123

124

2. Experimental

125 2.1. *Samples*

126 The samples used in this study are listed in Table 1 and their field relations are shown in Fig. S1
 127 Three of these samples are equivalent to those used by Durrani et al (1976), sunlit (70180),
 128 partially shadowed (72321), and continuously shadowed (76240) regolith samples while the
 129 fourth is the sample from near the bottom of the 3 m drill core near the sunlit sample (70001).
 130 Available information on the samples have been compiled by Meyer (2007; 2010a; 2010b;
 131 2010c; 2010d).

Table 1. Samples used in this study with some background information.

Room temperature	Mass (mg)	Freezer	Mass (mg)	Description*	CRE age† (Myr)	I _s /FeO‡
70001,83	100	70001,84	100	Deep regolith	~485	~40
70180,8	100	70180,9	106	Sunlit surface regolith	~360	47
72321,41	100	72320,7	108	Partially shaded surface regolith	45-55	73
76240,45	100	76240,48	112	Permanently shaded surface regolith	~20	56

* Meyer (2007; 2010a; 2010b; 2010c; 2010d)

† Arvidson et al. (1975), Leich et al. (1975); Crozaz et al. (1974).

‡ Morris (1976, 1978); Morris et al. (1979)

132 70001. This sample was the lowest level of the Apollo 17 three-meter drill core which was
 133 collected in the middle of the valley, near the landing site (Fig. S1). Much of the considerable
 134 early work on the drill core was summarized by McKay et al. (1991).

135 The Apollo 17 deep drill core was irradiated on 22 December 1972 using medical X-ray
 136 equipment facilities at JSC (Duke and Nagle, 1976). The dose absorbed was not reported, but a
 137 typical dose for the routine medical X-ray is about 1 Gy (Mettler et al., 2018), enough to fill the
 138 lower temperature traps, say those corresponding to peaks between 100-250 °C in the glow
 139 curve. Since these peaks are absent or weak in 70001, we assume that in ~50 years these peaks
 140 have decayed.

141 Schmitt et al. (2017) estimate that this sample of the core is regolith ejecta that was exposed to
 142 external solar proton and cosmic ray radiation and impact gardening for ~387 Myr after
 143 deposition at ~3.4 Ga. In addition, there has been continuous internal alpha and beta particle
 144 radiation from U, Th and K decay. Prior to deposition, there also was an unknown frequency of

145 periods of solar proton and cosmic ray radiation and impact gardening for the ~500 Myr since
146 regolith began to form on the surface of the valley floor. After deposition, the sample was
147 initially exposed to 387 Myr to a mean diurnal temperature of 214° K (~100° to ~375° K)
148 (Langseth, et al., 1973) and then, at ~3.0 Ga, it began to be progressively buried by younger
149 regolith ejecta zones until at about 1.5 Ga its temperature stabilized at a constant ~257° K (17° C)
150 with more than 130 cm of regolith ejecta above it.

151 The mean-lives for these peaks we calculate below are consistent with this. Nevertheless,
152 artificial exposure through the use of CT scanning must be kept in mind when discussing the
153 radiation history of extraterrestrial samples from JSC and several major museums (see Sears et
154 al., 2016; 2018).

155 *70180*. Regolith sample 70180 was collected on the surface near the drill core site (Fig. S1).
156 Being a surface sample 70180 has suffered the full range of alteration, thermal cycling,
157 micrometeorite bombardment, cosmic ray exposure, and gardening. In addition, solar protons
158 and alpha radiation from internal sources (U, and Th) have caused reduction of Fe²⁺ to Fe⁰
159 (Schmitt, 2022) producing an Is/FeO maturity index of 56 (Morris, 1976).

160 *72320 (and 72321)*. These samples were collected about 20 cm under the E-W overhang of
161 Boulder 2 at Station 2 at the base of the South Massif (Fig. S1). On the basis of its intermediate
162 natural TL compared to 70180 and 76240, Durrani et al. (1976) suggested that 72320 was only
163 partially shaded, consistent with the astronauts' description during collection. The roughly N-S
164 orientation of the overhang also indicates that morning sun would illuminate the otherwise
165 largely shadowed area. The fully exposed top of the shadowing boulder has a cosmic ray
166 exposure age of 52 ± 1.5 Myr (Leich, et al., 1975)

167 *76240*. This sample was from continuously shadowed soil from about 70 cm into a shadowed
168 overhang under Boulder 4 at Station 6 at the foothills of the North Massif (Fig. S1). The sample
169 came from the top 5 cm of the shadowed surface. Durrani et al. (1976) estimated that the shadow
170 formed 40-60,000 years ago. However, as will be evident below, this value is a minimum age
171 since the natural TL is in an equilibrium state. The maturity indexes of 76240 and other regolith
172 samples at Station 6 strongly suggest that the shadow formed when the boulder came to rest and

173 broke into several fragments between the boulder's reported cosmic ray exposure ages of ~20
174 Myr (Cozaz, et al., 1974).

175 *2.2 – 2.4. Experimental details*

176 Our methods for TL measurement are described in the supplement Sections S2.2 to S2.4. These
177 include how we make a correction for black body radiation, how we fit theoretical curves to the
178 observed glow curves, and how we estimate activation energies for the first peak in the glow
179 curve using the traditional initial rise method.

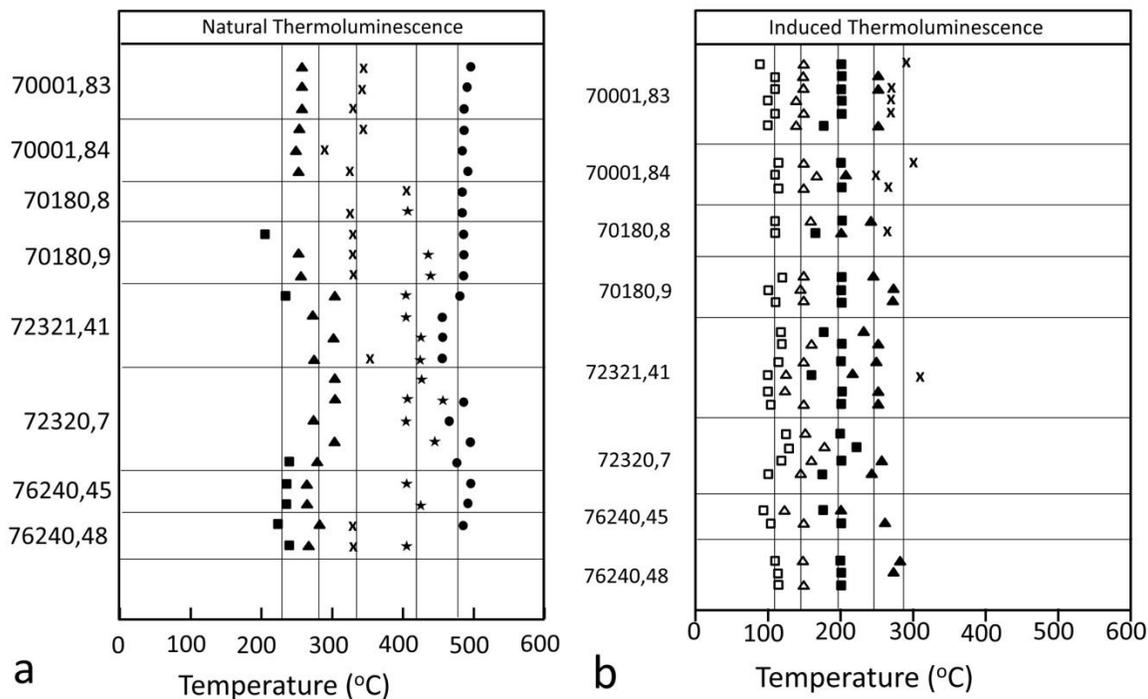
180 **3. Results**

181 *3.1. Visual Inspection of the glow curves*

182 To provide ground truth for more sophisticated glow curve analysis we first performed a visual
183 inspection of the glow curves to locate peaks and estimate their approximate relative intensities.
184 Plots of the peak positions for natural and induced TL are shown in Fig. 1. Table S1 lists peak
185 positions and peak intensities for natural TL. Table S2 lists the same data for induced TL.

186 As expected the natural TL for the room temperature and freezer samples are very similar.
187 Average peak positions for samples in their natural state are 223 ± 15 , 270 ± 18 , 339 ± 29 , 413 ± 14 ,
188 475 ± 9 °C. The glow curves for the irradiated samples show new peaks at 108 ± 3 , 147 ± 2 and
189 192 ± 2 °C. We number these peaks 1-8 in order of increasing glow curve temperature.

190 The intensity of the TL in all our samples, natural and induced, is very high, ranging from about
191 1000 cps to about 50,000 cps. (For comparison the Dhajala meteorite, which is often used as a
192 laboratory standard for TL studies, produces about 40,000 cps at its major peak.) For the natural
193 TL samples the peak at 475 ± 9 °C which extends beyond 500 °C and is always the strongest while
194 for the induced TL curves the three lowest temperature peaks are the strongest.



195

196 *Fig. 1. (a) Visual representation of the peaks and inflections (indicative of peaks) for the present Apollo 17*
 197 *samples. "Temperature" refers to the temperature at which the peak appears as the samples are heated. The*
 198 *vertical lines are means. Left, natural TL, mean ± 1 sigma, are 223 ± 15 , 270 ± 18 , 339 ± 29 , 413 ± 14 , 475 ± 9 °C. (b)*
 199 *TL peaks and inflections present after irradiating the samples with ^{90}Sr beta radiation, mean ± 1 sigma, are 108 ± 3 ,*
 200 *147 ± 2 , 192 ± 2 , 243 ± 24 , 289 ± 43 °C. As much as half of the 475 ± 9 °C peak actually lies beyond the range of our*
 201 *equipment.*

202 3.2 Determination of E (and s) by the initial rise method

203 Estimates of the trapping depth E (in eV) of the first peak in the glow curve are given in Table 2.

204 We also indicate in Table 2 the glow peak to which the data apply, i.e. the first significant peak

205 identified by the curve fitting results. We also indicate s values calculated from E using Eq. S4.

206

Table 2. Values of E (eV) determined by the initial rise method, corresponding s values, and peak temperatures, T_p . * †

Sample*		Room Temperature Samples	Freezer Samples	Irradiated samples Room Temperature & Freezer Samples
70001, 83/84	T_p	~220 °C (peak 4)	~220 °C (peak 4)	~100 °C (Peak 1)
	E	0.98±0.20	1.18±0.08	0.95±0.06
	s	3×10^9 ($2 \times 10^7 - 4 \times 10^{11}$)	4×10^{11} ($6 \times 10^{10} - 2 \times 10^{12}$)	4×10^{12} ($6 \times 10^{11} - 2 \times 10^{13}$)
70180,8/9	T_p	~420 °C (Peak 7)	~420 °C (Peak 7)	~100 °C (Peak 1)
	E	1.03±0.01	1.05±0.31	0.86±0.11
	s	1×10^{10} ($7 \times 10^9 - 1 \times 10^{10}$)	2×10^{10} ($8 \times 10^6 - 3 \times 10^{13}$)	2×10^{11} ($7 \times 10^9 - 9 \times 10^{12}$)
72321,41/ 72320,7	T_p	~265 °C (Peak 5)	~265 °C (Peak 5)	~100 °C (Peak 1)
	E	1.17±0.31	1.03±0.06	0.92±0.05
	s	3×10^{11} ($2 \times 10^8 - 7 \times 10^{14}$)	9×10^9 ($2 \times 10^9 - 4 \times 10^{10}$)	1×10^{12} ($3 \times 10^{11} - 6 \times 10^{12}$)
76240,45/48		~220 °C (peak 4)	~220 °C (peak 4)	~100 °C (Peak 1)
	E	1.11±0.07	1.15±0.06	0.87±0.03
	s	7×10^{10} ($1 \times 10^{10} - 4 \times 10^{11}$)	2×10^{11} ($5 \times 10^{10} - 7 \times 10^{11}$)	3×10^{11} ($2 \times 10^{11} - 8 \times 10^{11}$)
<i>Mean ± sigma</i>	E	1.08±0.07	1.10±0.06	0.90±0.02
<i>Mean</i>	s	3×10^{10}	6×10^{10}	8×10^{11}
<i>(± 1s range)</i>		($6 \times 10^9 - 1 \times 10^{11}$)	($1 \times 10^{10} - 2 \times 10^{11}$)	($4 \times 10^{11} - 1 \times 10^{12}$)

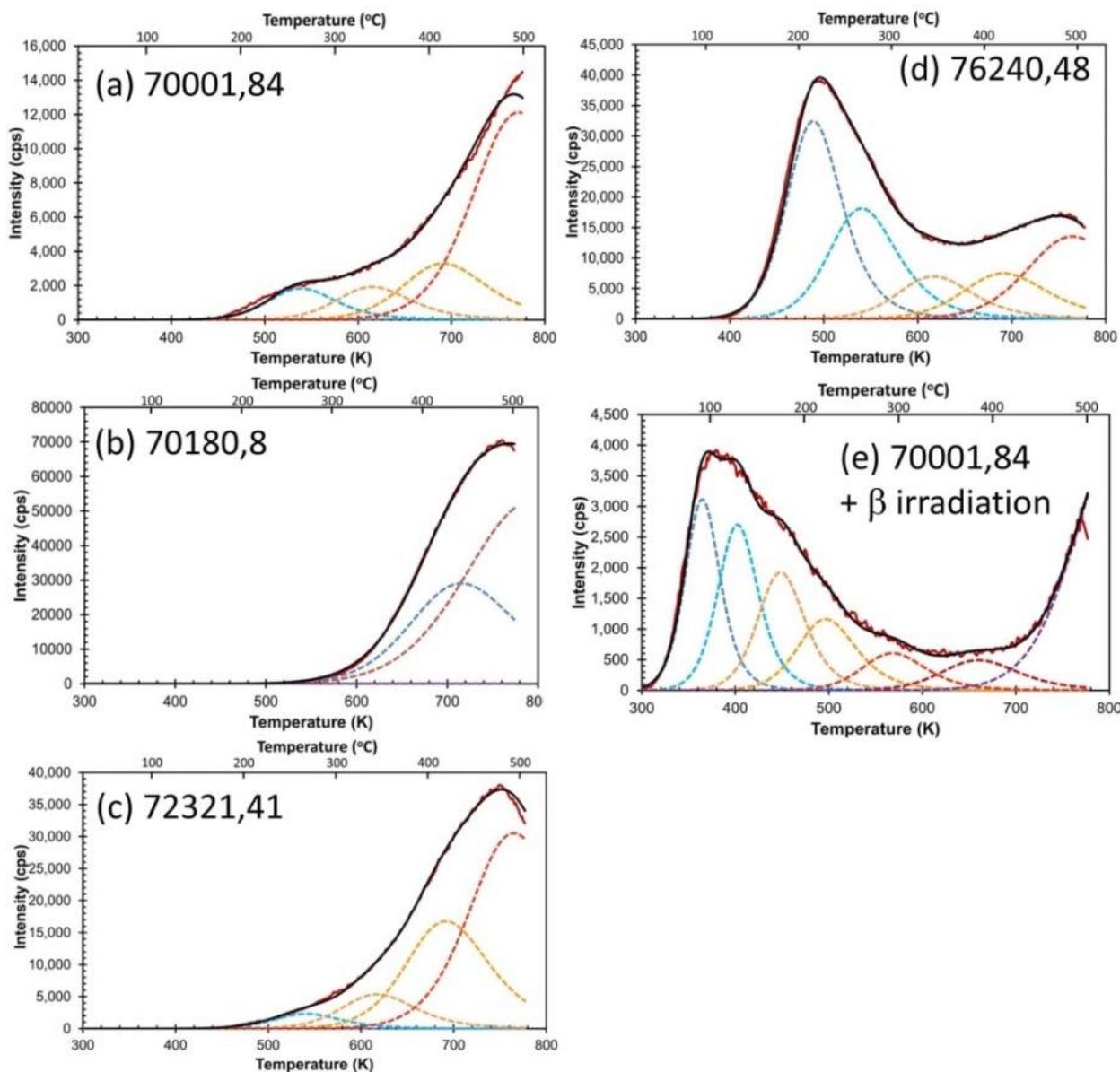
* T_p refers to the peak to which these data apply as judged from Fig. 2.

† s has been calculated from E assuming peak temperatures of 225 °C for natural curves and 100 °C for induced curves and a heating rate of 7.5 °C/s. The range in parenthesis refers to the values calculated for ±1s.

207 3.3. Curve fitting to natural TL curves

208 As discussed briefly in the Introduction, the TL of silicates is not emitted at a single heating
 209 temperature but over a broad range of temperatures and in the form of a number of overlapping
 210 peaks, each with its own value for E and s. The size of each peak, i.e. the number of electrons
 211 trapped at each defect, is governed by the kinetics of build-up due to ionizing radiations and
 212 thermal decay. Radiations are typically in the keV to GeV range and it is normally assumed that
 213 the ionization fills each trap according to their individual cross sections for electron capture. A
 214 relationship, proposed by Schmitt (2023), between trap filling and type and energy of radiation
 215 has yet to be experimentally explored.

216 Our procedure for fitting theoretical curves to the observed glow curve in order to identify and
 217 utilize the individual peaks is described in Section S2.4 and examples appear in Fig. 2. Our
 218 values for the activation energy, E , and rate constant, s , for the five peaks in the natural TL
 219 curves were the result of curve fitting to over 100 glow curves and are given in Table 3.



220

221 *Fig. 2. Examples of curve fitting for samples in their natural states (Fig. a-d) and a sample that*
 222 *has been drained of its natural TL and given a test dose of beta radiation (Fig. e). The induced*
 223 *curves (Fig. e) look essentially the same for all samples. The individual peaks identified by the*
 224 *curve-fitting process are indicated with broken lines.*

225 The curve fitting procedure does not yield uncertainties for the individual parameters but instead
 226 the software determines a measure for the quality of the fit between the theoretical and observed
 227 glow curve. For the natural glow curves all yielded a sigma of better than 0.5% which is
 228 consistent with the visual impression that the fits are excellent.

229 3.4. *Curve fitting to induced TL curves*

230 From curve fitting to over 300 glow curves our values for seven peaks in the induced TL curves
 231 are also given in Table 3. An example is shown in Fig. 2e. The fits obtained were not as good as
 232 for the natural curves but still acceptable with a 1σ goodness-of-fit of 1.0-1.5%. The main cause
 233 of the poorer fit was the higher temperature peaks which are sometimes weak or absent in the
 234 induced curves. Peaks 6 and 7 could not be resolved in the induced curves, although this was not
 235 a problem for the natural curves. In addition, the 500 °C peak is only partially sampled since our
 236 equipment cuts-off at this temperature. This explains the discrepancy between the induced and
 237 natural E values for peak 8. Otherwise the values for natural and induced curves agree within
 238 experimental uncertainties. We note that s values differ by factors of 2 to 7 which we consider
 239 good agreement, since it is within an order of magnitude.

Table 3. Selected E, s and T_p values for natural and induced glow curves and corresponding mean lives.*

Natural TL							
Peak number	4	5	6	7	8		
E (Ev)	0.95	0.94	1.13	1.26	1.39		
s (s^{-1})	3.83×10^8	2.83×10^7	7.91×10^7	5.39×10^7	3.24×10^7		
T_p (°C)	216	268	344	419	492		
Induced TL							
Peak number	1	2	3	4	5	6 and 7*	8†
E (Ev)	0.78	0.85	0.92	0.92	1.13	1.26	1.53
s (s^{-1})	9.95×10^9	5.13×10^9	1.85×10^9	1.41×10^8	5.83×10^8	1.61×10^8	6.11×10^7
T_p	92	130	175	223	295	387	498

* Comments

- Peaks 1 and 2 are absent in the natural curves. Peak 8 (at 492 °C) is a composite of natural and induced TL since a single heating to 500 °C removes only half the peak. Data for this peak should be treated with caution
- Peaks 5-8 in the induced curves are problematic because they are so weak. Peaks 6 and 7 are not resolved.
- For natural and induced curves the peak at 498 °C is a composite of natural and induced TL since a single heating to 500 °C removes only half the peak. Data for this peak should be treated with caution.

240 3.5 *Total counts in the peaks by curve fitting.*

241 Table S3 summarizes the n values (total number of counts, i.e., the area under each peak) we
 242 obtained from our curve fitting procedures, expressed in millions of counts. The number of

243 counts in the natural curves varied from essentially zero to 7 million while the number for the
 244 induced curves varied from zero to about 3 million. In both cases the largest number of counts
 245 was observed for peak 8. In the natural curves peaks 1, 2 and 3 were always absent. The ratio of
 246 the sigma to the mean is about 0.28 for all peaks in the natural curves and about 0.25 for peaks 1-
 247 6 in the induced curves. For peak 7 in the induced curves this ratio is around 0.40 reflecting the
 248 known difficulties in measuring this peak.

249 4. Discussion

250 *4.1. Comparison of the results of the visual inspection of the glow curve with the curve fitting* 251 *results – validating the curve fitting technique*

252 Having shown that the ~220 °C peak is behaving as expected and the comparisons we have made
 253 using simple peak heights yields reasonable values for E and s for this peak, we now wish to
 254 perform a more detailed analysis of the glow curves with the aim of (1) understanding the TL
 255 characteristics of lunar regolith and (2) quantify the properties of the TL glow curve, that is the
 256 number of peaks, their E and s values, and the intensity of each peak. An important element of
 257 our approach is curve fitting. This has not previously been attempted with lunar samples and we
 258 expect regolith to be particularly challenging in view of its heterogeneous nature although the
 259 gardening process during exposure at the surface homogenizes most characteristic parameters,
 260 such as maturity index (Is/FeO), composition, and cosmic ray exposure. We will therefore
 261 establish some ground truth by comparing data obtained by visual inspection of the glow curve
 262 with data obtained by curve fitting. The main characteristics of the glow curve are peak
 263 positions (which also means number of peaks) and peak intensities.

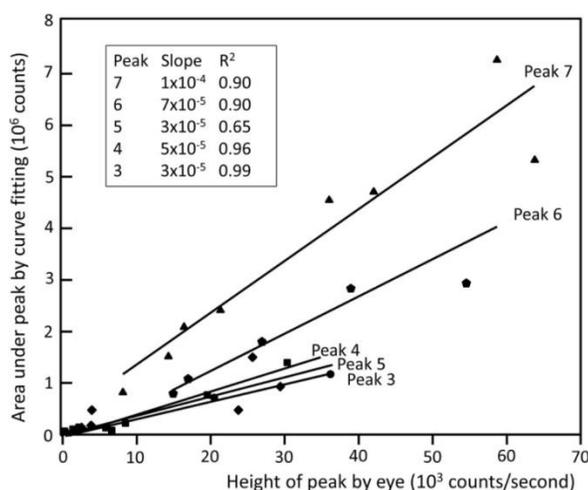
Table 4. Comparison of peak positions (°C) determined by eye and by curve fitting.*

	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8
Visual natural				223±15	270±18	339±29	413±14	475±9
Curve fitting natural				216	268	344	419	492
Visual induced	108±3	147±2	192±2	230±12	271±5			
Curve fitting induced	92	130	175	223	295	387		498
Nominal value	~100	~140	~180	~220	~265	~340	~420	~480

* Uncertainties for the visual data are one sigma based on replicate measurements. Such uncertainties are not available for the peaks used in curve fitting (which are determined by the selected values for E and s) but the fits have an uncertainty of <5% so uncertainties on peak positions are probably on the order of 5%. Peaks 6 and 7 are not resolved in the induced curve fitting method because they are too weak.

264 *Peak positions.* The peak positions determined by eye (visual data) and by curve fitting are
 265 compared in Table 4. We can say that the data are consistent but not independent because peak
 266 positions determined by visual inspection guided the choice of peaks for curve fitting.
 267 Nevertheless, despite the uncertainties on the visual data sometimes bring quite large the
 268 consistency across methods is reassuring. These data are presented in visual form in Fig. S3. For
 269 future convenience, we also list “nominal” values in Table 4.

270 *Peak intensities.* Figure 3 is a plot of peak intensity measured directly from the glow curve
 271 against the number of counts in a peak determined by curve fitting. According to TL theory
 272 higher temperature peaks are broader than low temperature peaks but for each peak there is a
 273 linear correlation between the two parameters, suggesting no major errors in the curve fitting
 274 process.



275
 276 *Fig. 3. Plot of number of counts in a peak (from curve fitting) against peak height (from visual*
 277 *inspection) for the five peaks in the TL glow curve. Each peak shows a positive correlation but,*
 278 *as expected from TL theory, the slope is steeper for higher temperature peaks. Peaks 1 and 2*
 279 *and not shown for clarity but plot among the data for peaks 3-5.*

280 **4.2. Selected (preferred) values for E and s , and mean-lives**

281 By comparing the results obtained with the natural samples, for the induced TL samples and the
 282 results obtained by the initial rise technique (see Table S5 for details) we suggest that (1) E
 283 values have 2σ uncertainties of $\pm 5\%$; (2) s values are good to within a factor of two to three; (3)
 284 peak temperatures have 2σ uncertainties $\pm 5^\circ\text{C}$. This is consistent with the data obtained by eye
 285 (Fig. 1). We summarize our selected values in Table 5. We are told that the storage facilities at

286 JSC have been kept at 293 K since 1969, although brief power outages from hurricanes may
 287 have caused some warming. We have also included the mean life, τ , of each TL peak for freezer
 288 temperature (253 K), room temperature (293 K), and lunar daytime equatorial temperatures (380
 289 K which we have calculated from:

$$290 \quad \tau = s^{-1} \exp(E / kT) \quad (1)$$

291 where k is Boltzmann's constant and T is the environmental temperature (in K). In the next two
 292 sections of our paper we consider whether our decay observations for peak 4 of the 76240
 293 sample are consistent with our independent estimates of E and s .

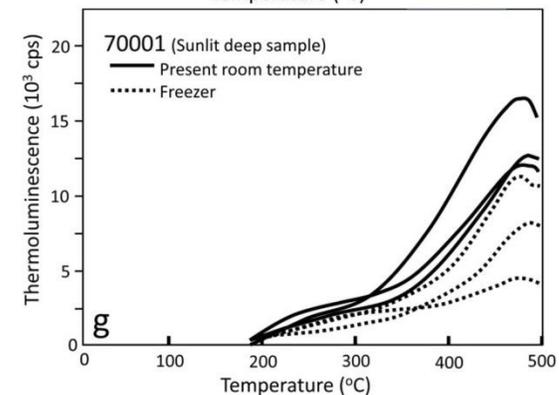
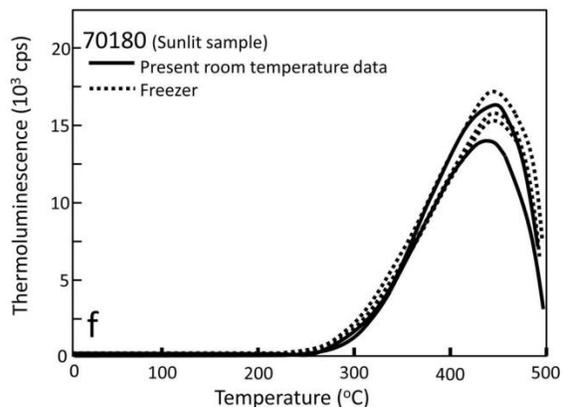
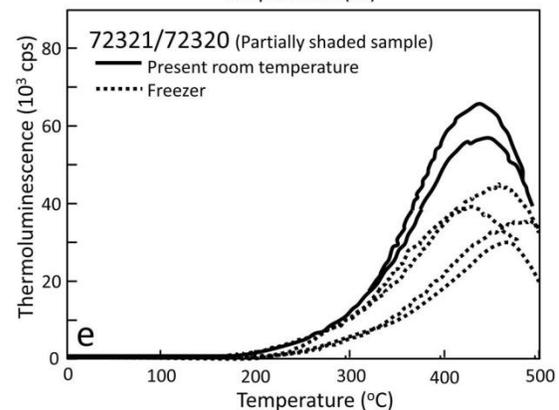
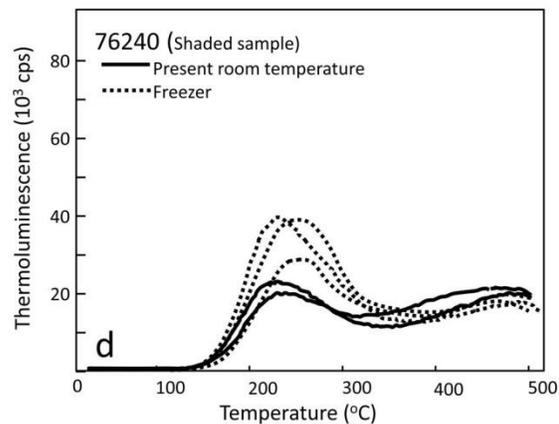
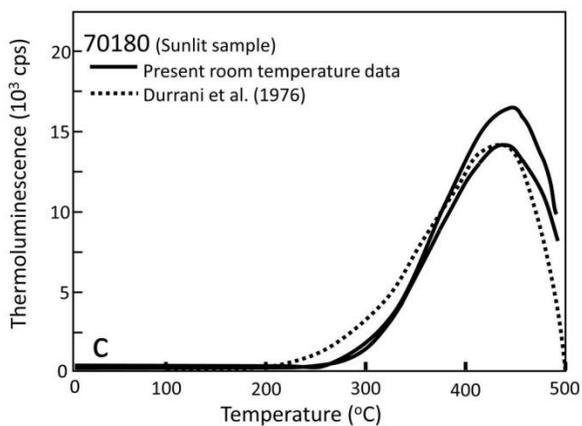
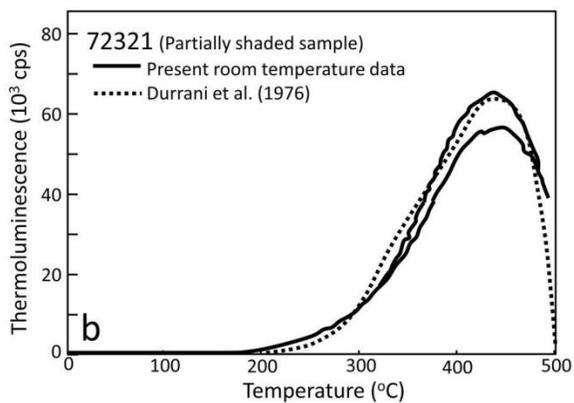
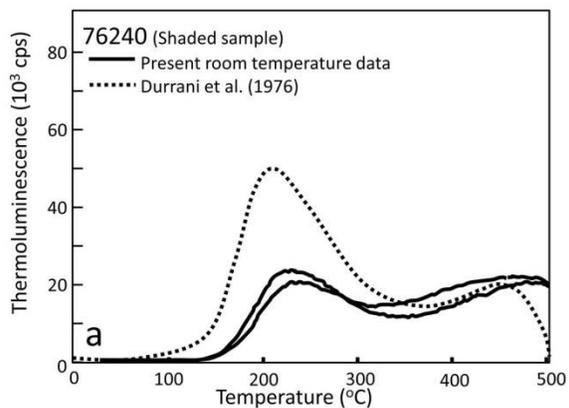
Table 5. Selected values for E and s based on Table 2 and Table 3 and calculated mean lives calculated from Eq (1)..

	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6	Peak 7	Peak 8
E (eV)	0.85	0.85	0.92	1.1	1.2	1.1	1.2	1.4
s (s^{-1})	10^{11}	5×10^9	2×10^9	10^9	10^{10}	8×10^7	10^8	3×10^7
τ (253 K)	10 day	198 day	34 yr	259,490 yr	3 Myr	3 Myr	255 My	8,189 By
τ (293 K)	70 min	23 hour	39 day	265yr	1389 yr	3,308 yr	139,000 yr	1,276 Myr
τ (380 K)	2 sec	38 sec	13 min	51 day	10 day	56 day	3200 day	3900 yr

294 **4.3. Comparison of glow curves from 1976 and now – the 46-year experiment**

295 Comparing our data with the earlier data is not straight-forward because Durrani et al. (1976)
 296 reported light intensities in “arbitrary units” so absolute comparison is not possible. They also
 297 used different arbitrary units for each sample. We can side step this issue by comparing glow
 298 curve shapes rather than absolute values. This assumes that all glow curve peaks are caused by
 299 the same mineral in basalts but it is usually a safe assumption (Batchelor et al., 1997; Akridge et
 300 al., 2004), although minor contributions from silica (our unpublished observation) and apatite
 301 have occasionally be observed (Sears et al., 2021). Second, the earlier authors used a slower
 302 heating rate (3.6 °C/s) than ours (7.5 °C/s), however this is a minor effect that should not be
 303 significant for this first look at the data.

**TL GLOW CURVE
COMPARISON**
Left column: 1976 vs now
Right column: 20 °C vs freezer



305 *Fig.4. Left column: Comparison of glow curves for Apollo 17 samples collected in 1976*
 306 *(Durrani et al., 1976) with data collected in 2022 for the samples stored at room temperature*
 307 *(20°C). The left hand axis applies to the present data; previous authors used arbitrary units so*
 308 *here we compare curve shapes rather than intensities. The previous authors did not include*
 309 *70001. Right column: Comparison of glow curves for Apollo 17 samples stored at room*
 310 *temperature with those stored in a freezer at -20°C. Note c, f, and g have a different y-axis.*

311 We compare the earlier data with the present glow curves in the left column of Fig. 4. We
 312 observe that the shaded sample (76240), with its strong peak in the ~220 °C region of the glow
 313 curve, appears to have faded by about 60% (Fig. 4a). We are comparing real data with a sketch,
 314 but if we assume the uncertainties on the sketch are comparable to ours, then the decay is
 315 $60\pm 10\%$. This decay corresponds to a mean-life of about 55 ± 9 years. Based on our present
 316 estimates for E and s, this peak (peak 4) has a mean-life of 50 years at 293 K. A $\pm 5\%$ error in E
 317 yields a range of 7 to 365 years, while an error of a factor of three in s yields a range of 17-168.
 318 In other words, considering the uncertainties in our data 50 years is in excellent (perhaps
 319 fortuitous) agreement with 60% decay.

320 We can actually use the observed decay measurements to calculate either E or s if we know the
 321 other. If we take our preferred value for E from Table 5 (1.10 eV) and substitute into Eq. (1) we
 322 get $s = 1.33 \times 10^9 \text{ s}^{-1}$, which is in excellent agreement with our preferred value of s in Table 5 of
 323 10^9 s^{-1} . Conversely, if we substitute our preferred value for s in Eq. (1) we calculate a value of
 324 1.01 eV which agrees within error of our preferred value. In other words, despite the rather large
 325 uncertainties on our independently determined values for E and s, they agree very well with the
 326 results of the fifty year experiment and the decay observed for the 220°C TL peak of 76240.

327 The situation is very different for 72320/1 and 70180; Durrani et al. (1976) did not include
 328 70001 in their study. The first peak for the sample from the partially shaded area (72320/1) is
 329 peak 5 (Table 2) with a mean life at room temperature (293 K) of 1389 years (Table 5). With a
 330 5% error in E or a factor of three uncertainty in s the range is 192 to 10,000 years. The expected
 331 decrease in TL intensity over fifty years is 3.5% and it is unlikely we could have detected this
 332 given the sample heterogeneity. The first natural peak in the glow curve of the sunlit sample
 333 (70180) is peak 7 with a mean life at room temperature of 139,000 years. With a 5% error in E
 334 or a factor of three error in s, the range of uncertainty is 19,000 to 1.0 million years. Thus we

335 expect to see no change in the natural TL of this sample over fifty years (the calculated value is
336 (0.04%) and this is our observation

337 **4.4. Compare room temperature and freezer samples:**

338 The glow curves comparing the present room temperature samples with the sample kept in a
339 freezer for almost fifty years are shown in the right side of Fig. 4 (i.e. Fig. 4d-g). Since all
340 samples were measured on the same apparatus we do not need to normalize in any way.

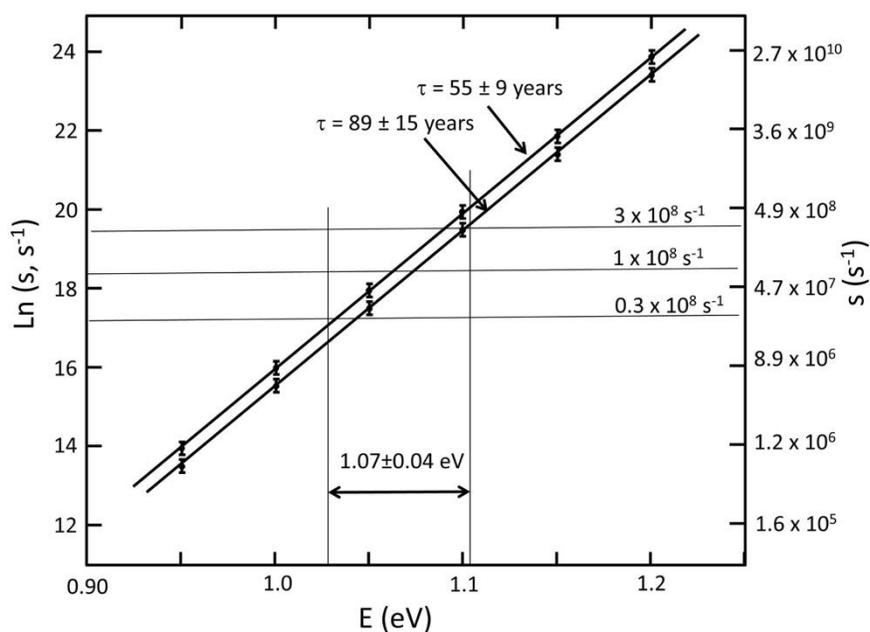
341 There is scatter in the glow curves for some samples, this is especially true of the freezer samples
342 for 72320 and 70001, and this can be explained by sample heterogeneity (as well as by vastly
343 different lunar surface exposure histories) as can the differences between room temperature and
344 freezer samples. Induced TL is a reflection of sample heterogeneity, not being affected by
345 radiation and thermal history. As an example, the mean induced TL peak height for 70001 room
346 temperature samples is 6100 counts while for the freezer samples it is 2200. By the same token,
347 the induced TL of the three 70001 freezer samples are 2300, 2250, and 1800, which explains the
348 scatter in these three glow curves. In summary, the scatter is easily explained by sample
349 heterogeneity, given potential variability in mineral frequencies between the small sample sizes?
350 We conclude that 72320/72321, 70001 and 70180 show no significant difference in their glow
351 curves. This is to be expected because of the long mean lives (Table 5). Using our preferred
352 values for E and s, the mean life of peak 4 at freezer temperatures is 26,430 years. If we allow
353 for a 5% uncertainty in E and a factor of 3 uncertainty in s, the range of possible mean lives is
354 2640 to 2.5 million years. The other peaks present in these samples have even longer mean lives
355 (Table 5). Thus our E and s values are consistent with our observation that the peaks observed in
356 natural TL curves for these samples are perfectly stable when the samples are stored in a freezer.

357 None of these arguments apply to the 220°C peak of 76240 which reflects a true difference in
358 thermal history for the *in situ*, freezer and room temperature samples. For 76240 the room
359 temperature value is 43% lower than the freezer value (20.38 ± 1.24 compared with 35.83 ± 5.92 ,
360 Table S1), or 43 ± 8 % including uncertainties. This is similar to the value obtained for the then-
361 and-now comparison of 60 ± 10 %, especially bearing mind that the old data is in the form of a
362 sketch. The mean life for the freezer vs room temperature difference is 89 ± 15 years. As
363 described earlier, the room temperature mean life predicted by our independent determinations of

364 E and s is 50 years with a range of 7 to 365 years assuming an uncertainty in E of 5% and a
 365 factor of 3 for the uncertainty in s.

366 4.5. Estimating E or s directly from the decay observations?

367 NASA's fifty year experiment yields two results for the decay of peak 4 of the 76240 sample; the
 368 then-and-now experiment yields a mean life of 55 ± 9 years and the freezer vs room temperature
 369 experiment yields a mean life of 89 ± 15 years. These data do not alone enable us to determine
 370 the kinetics of this peak, but it does allow some check on our estimates of E and s by other
 371 means. Figure 5 plots Equation 1 for the mean-lives observed here. If we accept an s value of
 372 10^8 s^{-1} (Table 5) and assume an uncertainty of a factor of three in either direction we find that E
 373 is $1.07 \pm 0.04 \text{ eV}$. This is in agreement with the independent laboratory determination of E using
 374 the initial rise method which is $1.08 \pm 0.07 \text{ eV}$ (room temperature samples) and $1.10 \pm 0.06 \text{ eV}$
 375 (the freezer samples) (Table 2).



376
 377 *Fig. 5. Constraining E and s values using the mean-life observations reported here for the*
 378 *shaded sample 76240. Assuming $s = 10^8 \text{ s}^{-1}$ with an uncertainty of a factor of three (based on*
 379 *independent measurements), the trap depth of peak 4 is found to be $1.09 \pm 0.06 \text{ eV}$ in agreement*
 380 *with the laboratory estimates using the initial rise method.*

381

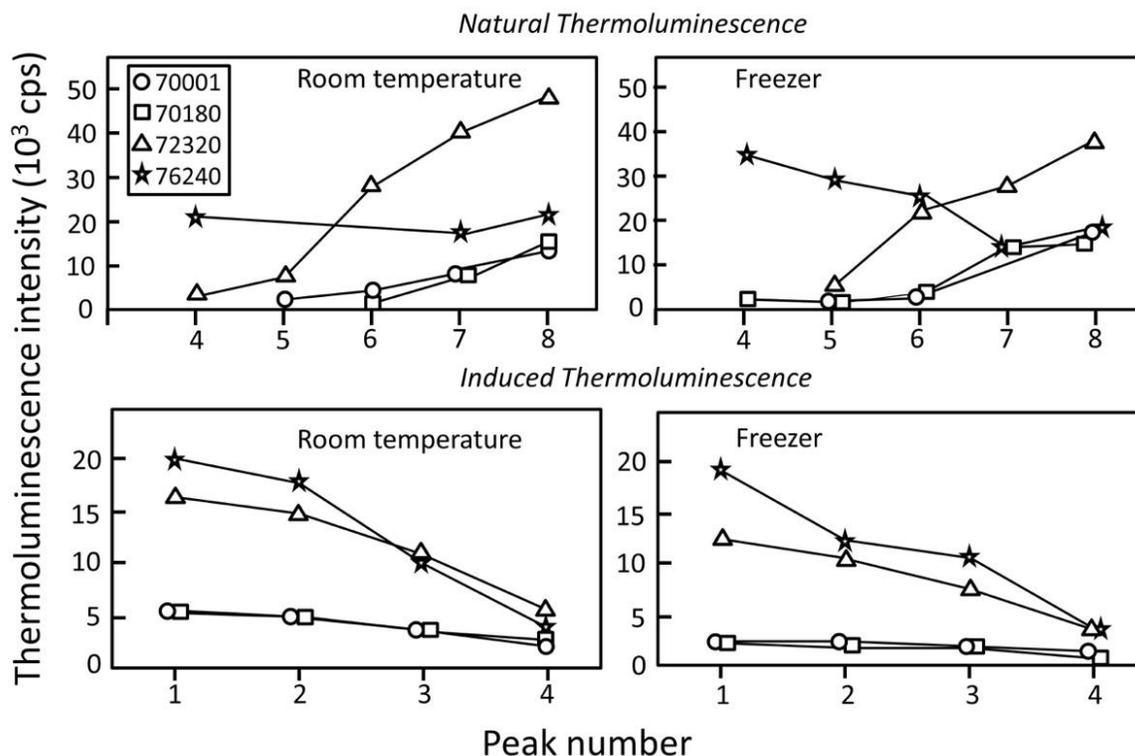
382

383 **4.6. *Induced TL and sample heterogeneity***

384 A discussion of the induced thermoluminescence properties of these samples and the remainder
385 of the Apollo 17 deep drill core appears in a companion paper (Sehlke and Sears, 2023).
386 Essentially, the valley samples have induced TL similar to Maria samples while the samples
387 from the foot hills of the massifs have highland values, a factor or five or so higher than the Mare
388 samples. This is the case even given that the composition of the valley samples indicate
389 significant mixing of the two types of regolith, ~30% Sculptured Hills regolith in valley sample
390 70180, for example. This difference can be attributed to different feldspar abundances. Some
391 petrographic data for our samples is given in Table S4. These data are in agreement with the
392 study of Batchelor et al. (1997). As one would expect of regolith samples there is a high degree
393 of heterogeneity as evident from the uncertainties and the induced curves for the freezer samples
394 76240 (Fig. 4). This heterogeneity would be increased in the comparison of small samples.

395 **4.7. *Total peak intensities (total counts per peak)***

396 Finally we compare the natural TL of samples stored at room temperature with the samples
397 stored in a freezer using the n values (i.e. total counts in each peak) determined by curve fitting
398 (Fig. 6). Error bars are not shown in the figure to avoid cluttering but are given in Table S3
399 (natural TL) and Table S4 (induced TL). We observe: (1) peak 4 in the 76240 sample (the peak
400 that was of primary interest to Durrani et al. (1976) is lower for the room temperature data than
401 for the freezer data by about 50%. This is in agreement with the peak shape observation
402 described earlier (Fig. 4); (2) except for 76240, the room temperature and the freezer values for
403 the natural TL are very similar; (3) as expected the induced TL data for the room temperature
404 and freezer samples are very similar; (4) 76240 and 72320 have the natural and induced data
405 which are higher by a factor of four or more than the Mare samples 70001 and 70180. This
406 reflects the highland nature of 76240 and 72320 and with their relatively high abundance of
407 feldspar; (5) We also know that there is very little overlap in the peaks present in the induced and
408 natural TL data, just peak 4 and the scatter shown in the induced data for peak 4 is comparable
409 with the analytical uncertainties, thus removal of the effects of heterogeneity by normalizing
410 natural data to induced data is not possible.



411
 412 *Fig. 6. Plots comparing the thermoluminescence intensity of the individual peaks (n values,*
 413 *areas under the peaks) determined by curve fitting. Peaks 1-3 are absent in the curves for*
 414 *samples in the natural state while peaks 5-8 are too weak to accurately measure for samples the*
 415 *which have had their natural signal removed and have been given a standard radiation dose in*
 416 *laboratory.*

417 The reason for the difference between peaks 7 and 8 in the room temperature and freezer samples
 418 is unclear. The room temperature curves resemble the published sketch of the glow curve of
 419 76240 by Durrani et al. (1976), given the thermal drainage of peak 4. However, we would
 420 expect peaks 7 and 8, which are thermally stable to be similar for the room temperature and
 421 freezer samples, especially given the similarity of the induced TL. Contamination by alien
 422 material is not a likely explanation for the difference, given the similarity of the induced curves.
 423 Instead, the contaminant material would have to have the same composition as the rest of the
 424 sample but a different (i.e. lower dose) radiation history. We are not clear how this could
 425 happen. We doubt very much that the radiation exposure in metal cabinets and the freezer the
 426 JSC laboratory was different. There are probably minor minerals such as quartz and apatite (or
 427 whitlockite) present in the samples but our experience is that they have very different glow curve
 428 shapes.

429 These data demonstrate that while much of the behavior of lunar sample natural TL is
 430 understood, there remain anomalies that only further data and further work will resolve. This is
 431 not true of the presence or absence of individual peaks. We do not know what defect or impurity
 432 centers give rise to individual peaks, this is still a subject of active research field for solid state
 433 physicists interested in the ionic solids used in dosimetry. We are a long way from
 434 understanding these properties for silicate minerals and glass, although the spectroscopists have
 435 made a start (Geake et al., 1977). However, we now know that there are many TL peaks (eight
 436 between room temperature and 500 °C) and we have a reasonable idea of their E and s values.

437 ***4.8. Equilibration temperatures at Taurus Littrow***

438 Durrani et al. (1976) used the natural TL data for Apollo regolith to calculate storage
 439 temperatures (which we prefer to call equilibrium temperatures). The relevant relationship is:

$$440 \quad T_{\text{eq}} = (E/k) / \{ \ln [s R_{1/2} / 0.693 r (N/n - 1)] \} \quad (2)$$

441 Where E and s are the kinetic parameters, k is Boltzmann's constant, $R_{1/2}$ is the radiation dose
 442 required to half fill the traps, r is dose rate, and N/n is the reciprocal of the fraction of traps filled.
 443 We have eight TL peaks in the glow curves of the present samples starting at about 100 °C. In
 444 principle, and as argued by Hoyt et al. (1971), the lower temperature TL peaks should be
 445 building up (i.e. growing faster than they are decaying), middle temperature peaks should be at
 446 equilibrium, and the highest temperature peaks should be "saturated", (i.e. decaying so slowly
 447 that they have reached a state where all the traps are filled). ***In general, the equilibration***
 448 ***temperature of the lowest temperature peak that is at equilibrium will be the storage***
 449 ***temperature for the whole sample.*** The challenge is to find a way of knowing which peaks are
 450 at equilibrium and which peaks are not. One approach is to make reasonable assumptions and
 451 test them by comparing T_{eq} with independent surface temperature estimates. Figure S6
 452 summarizes some literature data in visual form.

453 On the surface of the Moon the external ? radiation dose rate as measured by Chang'E 4 at 45° S
 454 176° E is 0.116 Gy/year (Zhang et al. 2020), close to the 1960s estimated global value of 0.10
 455 Gy/year (Haffner, 1967). This value will be affected by latitude, surface composition, internal
 456 U+Th concentration, and local topography. The partial shielding effects of boulders will locally
 457 modify radiation dose rates at specific locations, for instance, but we expect dose rates to vary by

458 less than an order of magnitude. For example, the TL profile across the 60 cm slab of the
459 Estacado meteorite shows a variation of less than a factor of two (Sears, 1975).

460 Temperatures at the lunar surface range from -140 to 400 K (e.g. Bauch et al., 2014; see also Fig.
461 S6). Near the poles, of course, temperatures are as low as 20-40 K are to be expected in the
462 permanently shaded crater interiors. Of course, there are also burial depth effects, also
463 summarized in Fig. S6. Soon after arrival on the Moon the Pragyán rover found that the
464 temperature of the regolith at 69.37°S, 32.35°E. dropped from 333 K to 263 K in only 10 cm
465 (ISRO statement, 2023). Temperatures stabilize, however, at about 257 K in the Apollo 17 heat
466 flow probe (Langseth, et al, 1973).

467 *Sunlit surface sample (70180,8)*. If we assume that peak 8 is at equilibrium then using our
468 selected values for E and s given in Table 5, and allowing for an uncertainty of $\pm 5\%$ in E and a
469 factor of 3 in s then T_{eq} is 370 ± 10 K. This is in agreement with the TL estimate of 371 K by
470 Durrani et al. (1976), and surface probes (384 K, Langseth et al., 1973; and 377 K (Song et al.,
471 2017)). The surface temperature obtained remotely for the center of the valley and reported by
472 Bauch et al. (2014; see Fig S6a) is ~ 380 K. Peak 7 is also present in the glow curves of 70180
473 but it yields equilibration temperatures ~ 40 K lower suggesting that this peak is not at
474 equilibrium. We note in passing that in a case where the heating follows a sine wave, it is the
475 maximum temperature levels that dictate TL stability.

476 *Buried sunlit surface sample (70001,83)*. A few meters below the surface the temperature of the
477 lunar regolith remains constant. The models of Malla and Brown (2015) and Vasavado et al.
478 (1999) indicate that while the surface cycles between ~ 100 K to ~ 380 K a few meters below the
479 surface remains a constant ~ 250 - 257 K (Fig. S6b). Additionally there is a thermal gradient.
480 Subsurface probes placed by the Apollo 17 astronauts indicated that while the surface was at
481 ~ 240 K at the time of measurement, a few meters deep the recorded temperatures were ~ 270 K.
482 (Keihm and Langseth, 1975; see Fig. S6c). The TL glow curve for this sample contains peaks 5
483 and 6, in addition to peak 8 (peak 7 could not be resolved), which have equilibration
484 temperatures of 280 and 285 K respectively, both ± 10 K, significantly higher than subsurface
485 values measured with thermocouples. The difference might be that thermocouples yield real-
486 time temperatures; TL is recording a time-averaged value (maybe $\sim 10^4$ years, see below). The

487 low temperature edge of a ~650 cps plateau in the core (Sehlke A. and Sears, 1922) may reflect
488 peak 4.

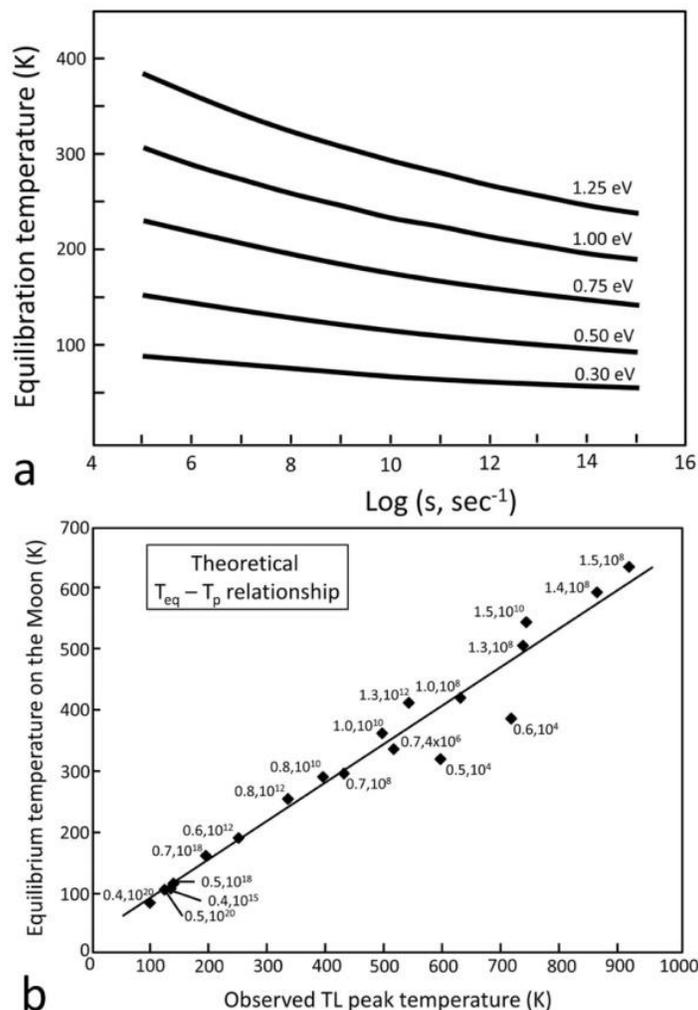
489 *Partially shaded sample (72321,41)*. The equilibration temperature for peak 5, the first strong
490 peak observed for this sample, is 280 ± 10 K, well below the value for the sunlit sample from the
491 middle of the valley and comparable with the 3 m deep sample. Durrani et al. (1976) did not
492 report a temperature for this sample, but we assume it would be between 371 K (their sunlit
493 sample) and 256 K (their shaded sample), although 256 is probably too high for the shaded sample
494 as discussed above. In this sense, our value is as expected.

495 *Fully shaded sample (76240,48)*. The first strong peak in this sample is peak 4 which yields an
496 equilibration temperature of 266 ± 10 K; the parameters for the trap indicate that at this
497 temperature build-up due to radiation and decay due to thermal draining are in balance. Our
498 value compares with the Durrani et al. (1976) estimate of 256 ± 15 K. Thus the difference in
499 temperature between the shaded and sunlit samples is on the order of 115 K. McGovern et al.
500 (2013) used the Diviner radiometer on the Lunar Reconnaissance Orbiter to compare the
501 temperatures of shadowed craters with adjacent planes Fig. S6d). These authors found a
502 temperature difference of 75 to 120 K between the sunlit and shadowed areas. Again our TL
503 data are consistent with other techniques.

504 **4.9. Relationship between T_{eq} and $\log s$ and E**

505 With current interest in water ice and the exploration of the lunar South Pole, we need to
506 consider TL glow curves well below room temperature. Figure 7a looks more closely at the
507 relationship between E , s and equilibration temperature. Very small values of s , say 10^4 - 10^6
508 produce equilibration temperatures that are strongly dependent on E being around 600 K for trap
509 depths of 1 eV and 200 K for a very low trap depth of 0.3 eV. For the E and s values obtained
510 here for peak 3, ~ 1 eV and $\sim 10^8$ s⁻¹, a temperature of ~ 500 K is indicated, in good agreement
511 with Durrani et al. (1976). For much larger values for s , 10^{11} to 10^{13} say, the range of
512 equilibration temperatures is smaller and varies from 100 to 325 K. How these E and s values
513 relate to the peak temperatures is shown in Fig. 7b; this range of E and s values produced peak
514 temperatures over the full range of ~ 100 K to 1000 K.

515 The bottom line of Fig. 7 is that with plausible values of E and s we can expect to see
 516 thermoluminescence peaks with equilibration temperatures as low as 100 K, a temperature at
 517 which water vapor would condense to ice assuming the regolith was at this temperature for
 518 sufficient time (Schorghofer and Taylor, 2007).



519
 520 *Fig.7. (a) The dependence of the equilibrium temperature for a natural TL peak on E and s . (b)*
 521 *Theoretical relationship between equilibrium temperature for natural TL peaks and peak*
 522 *temperature in the glow curve. Indicated by each data point are the assumed E (eV) and s (sec^{-1})*
 523 *values.*

524 4.10. Storage temperatures for returned lunar samples

525 Durrani (1972) and others recommended that NASA keep some of its lunar samples in a freezer
 526 and they agreed. This study is a result. Several of our samples were from the open lunar surface

527 and had experienced temperatures of ~400 K and there was no advantage in freezing them.
 528 However, 76240 demonstrates that even rocks from the surface and at low latitudes (Apollo 17
 529 landed at 20° N, 31° E), albeit in the shade, display a natural TL signal that decays at room
 530 temperature over 10s of years. Durrani's decay and mean-life arguments were based on samples
 531 he had artificially irradiated and known to have peaks that rapidly decay at room temperatures.
 532 He was, in effect, arguing that should sample ever be returned from colder regions they will need
 533 to be kept in a freezer on arrival on Earth or during transport there. Of course this becomes more
 534 critical if samples are returned from the lunar poles. In fact, even storage in liquid nitrogen
 535 might not be sufficient to retain an unaltered signal for samples from permanently shaded regions
 536 around the South Pole.

537 Since TL apparatus is robust, low power, low data rate and low weight, these temperature
 538 problems would be circumvented by instruments designed to be used on the lunar surface, either
 539 remotely or hand-held (Sehlke and Sears, 2022).

540 **4.11. Time**

541 The presence of water in the polar regions of the Moon depends not just of temperature but on
 542 time; the water vapor must have sufficient time to accumulate even when temperatures are
 543 favorable. Durrani et al. (1976) used the equation:

$$544 \quad t = \emptyset / R \quad (3)$$

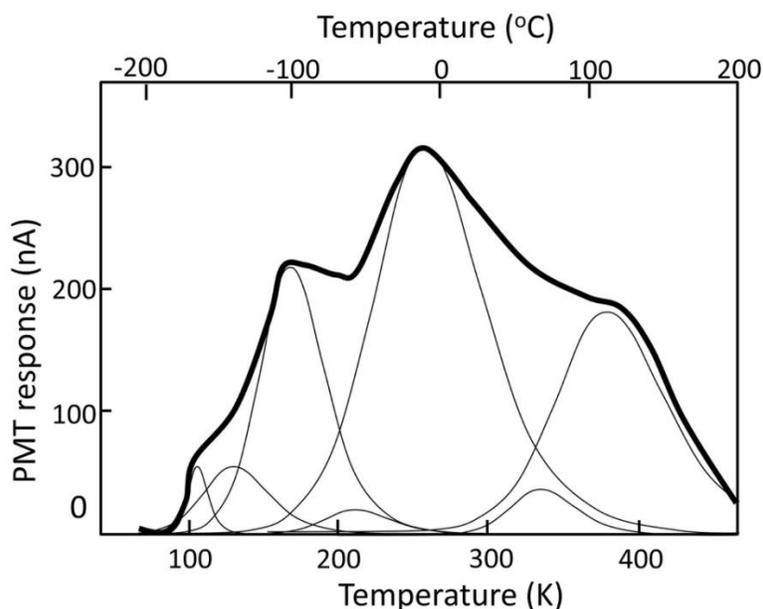
545 where t is time, \emptyset is dose (estimated from the natural TL using laboratory calibration), and R is
 546 the dose rate taken from Haffner (1967), which agrees closely with a recent lander measurement
 547 (Zhang et al., 2020). Durrani et al. (1976) obtained ages of 3.98×10^4 year for TL in the glow
 548 curve region 306-378 °C, which would be dominated by our peak 6, and 5.64×10^4 year for TL
 549 in the glow curve region 378-486 °C which encompasses our peaks 7 and 8. Peak 8 is subject to
 550 a number of difficulties because, like ours, their equipment cuts out at 500 °C. Durrani et al.
 551 (1976) assumed that these peaks were still growing when they considered these estimates as
 552 actual exposure ages; if the peaks were saturated (traps full) or at equilibrium (build-up equals
 553 decay) then they would be lower limits. Of course lower limits are also of value in determining
 554 the likelihood of water at a given cold trap.

555 In the supplement we calculate the non-equilibrium temperature at which mean-lives of the
556 relevant peaks are comparable to the cosmic ray exposure ages. At the moment we have no
557 scenario that would justify these comparisons but we hope this is the beginning of such a
558 discussion, which should also include I_s/FeO values.

559 *4.12. Prospecting for lunar cold traps*

560 The normal lunar temperature range at the equator is 140 K to 400 K. Temperature in polar
561 permanently shaded regions can be as low as 20-40 K (Sefton-Nash et al., 2019). Theoretical
562 calculations suggest that water would be trapped under lunar conditions at ~ 100 K (Watson, et
563 al., 1961; Arnold, 1971; Schorghofer and Taylor, 2007). From Fig. 7 we find that values of E in
564 the order of 0.4 eV with a frequency factor of about 10^{20} would have an equilibrium temperature
565 of ~ 100 K and correspond to a peak at 105 K in the TL glow curve.

566 Thus natural thermoluminescence with peaks in the vicinity of 100 K can serve as a
567 thermometer. What is required is a quantitative understanding of peaks with E values of ~ 0.5 eV
568 and s values in the order of 10^{18} to 10^{20} s^{-1} . However, knowing the temperature is not sufficient;
569 we also need to know how long the regolith has been at that temperature. Natural TL also
570 provides a means of determining time, as shown by Durrani et al. (1976). If the natural TL of a
571 100 K peak is known and using laboratory calibration is converted to dose, as is the practice in
572 conventional use of TL as a dosimeter, then dividing by dose rate, which is well known as will
573 be the internal concentration of U and Th, will give us the time during which the regolith was
574 accumulation a TL signal. Durrani et al. (1976) found ages in the range 10^4 - 10^5 years for peaks
575 the natural glow curves for Apollo 17 regolith samples; however, it is not clear that his technique
576 can be applied to lunar TL data. Considerations of maturity indices and boulder dynamics
577 indicate that the shadow for 76240 formed ~ 20 Myr ago. If the natural TL is at equilibrium then
578 this time will be a lower limit, if the natural TL is still building-up, then the age will be a true
579 age, although one must correct for decay during the buildup. Either way, it provides the answer
580 to whether the regolith has been at these low temperatures long enough to accumulate volatiles.



581
 582 *Fig. 8. Low-temperature TL glow curves for Apollo 15 regolith. Similar curves were obtained*
 583 *for Apollo 14 and 16 regolith samples. The heavy curve is the observed glow curve and the fine*
 584 *lines are our analysis in terms of individual peaks. (Blair et al., 1972a; sample A, 435-485 nm).*

585 Thermoluminescence glow curve curves for Apollo 14, 15 and 16 regolith samples at liquid
 586 nitrogen temperatures have been published by Blair and his colleagues (Blair et al., 1972a,b).
 587 An example of their glow curves is shown in Fig. 8. These authors suggested that the curves
 588 could be resolved into four peaks but we suspect that several weaker peaks are also present.
 589 Clearly, TL down to 100K is displayed by these samples which would be suitable for cold trap
 590 prospecting. However, there are several caveats to be mentioned in comparing Blair's glow
 591 curves with ours. Their samples were irradiated to 0.75 krad with 160 MeV protons, the heating
 592 rate during measurement was 0.8 °C/s and they mounted the samples for measurement with
 593 luminescent silicone grease. Thus detailed comparison is not justified, rather we show Fig. 7 to
 594 illustrate that low temperature measurements are possible and that TL in the 100 K region is
 595 present. We are also concerned that Blair et al. 1972b showed the same curve with a different
 596 temperature axis. Nevertheless with so many peaks in the room temperature to 500 °C region of
 597 the glow curve there will be as many down to very low temperatures. Sun and Gonzales (1966)
 598 published a glow curve for the meteorite Cumberland Falls from -150 °C to 250 °C which
 599 showed many peaks below room temperature. While the luminescent phase was enstatite, not
 600 feldspar, this does indicate the feasibility of other silicate minerals having multiple peaks with
 601 very low equilibration temperatures.

602

5. Conclusions

603 We have conducted the first major study of the natural thermoluminescence of Apollo regolith
604 samples using curve analysis as our major analytical technique. We have determined trap depths
605 (i.e. activation energies) and pre-Arrhenius factors (i.e. rate constants) for the five TL peaks in
606 natural samples and seven peaks in samples irradiated at room temperature with a laboratory
607 radiation source. The results compare favorably with those obtained by traditional independent
608 laboratory experiments.

609 The fifty-year experiment NASA organized for Apollo 17 samples, whereby some samples were
610 stored at room temperature and others at -20°C was successful. The sample of 76240 (the fully
611 or continuously shaded sample) stored at room temperature showed an approximately 50% decay
612 while the 76240 sample stored in the freezer show no measureable decay. This is consistent with
613 the kinetic parameters we have derived and means that samples returned from shaded areas at
614 any lunar latitude should be stored in a freezer if there is any possibility that studies of their
615 radiation history will be performed in the future.

616 The relationships that have been developed during this work suggest that lunar cold traps could
617 be located by astronaut-held or robotic thermoluminescence instrumentation. It seems that
618 simple developments would enable this technique to be used in a remote fashion so that samples
619 do not need to be handled.

620

6. Open Research

6. Open Research

622 *6.1 Data Availability Statement:* All experimental data, including glow curves and their
623 metadata from this study, are publicly available at (Sehlke, 2024) under the CC BY 4.0 (Creative
624 Commons Attribution 4.0 International) license.

625 *6.2 Software Availability Statement:* No software or models were used to generate the data
626 discussed in this manuscript. The data analysis and interpretation methods employed are fully
627 described in the Methods section of this manuscript for transparency and reproducibility.

628

629

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805 *ANGSA Science Team members. Names in bold indicate Project Science Group (PSG) members:*

Name	Email Address	Affiliation
Alex Sehlke	sehlke@baeri.org	NASA Ames Research Center
Derek Sears	dsears@uark.edu	NASA Ames Research Center
Jessica Barnes	jjbarnes@lpl.arizona.edu	University of Arizona
Carolyn Crow	carolyn.crow@colorado.edu	University of Colorado Boulder
Maryjo Brounce	mbrounce@ucr.edu	University of California Riverside
Jeremy Boyce	jeremy.w.boyce@nasa.gov	NASA Johnson Space Center
Jed Mosenfelder	jmosenfe@umn.edu	University of Minnesota
Tom Zega	tzega@lpl.arizona.edu	University of Arizona
Zoë Wilbur	zewilbur@email.arizona.edu	University of Arizona
Sean Pomeroy	Sean.Pomeroy@colorado.edu	University of Colorado Boulder
Iunn Jenn Ong	oij4869@email.arizona.edu	University of Arizona
Timothy Hahn	timothy.m.hahn@nasa.gov	NASA Johnson Space Center
Timmons Erickson	Timmons.m.erickson@nasa.gov	NASA Johnson Space Center
Jamie Elsila Cook	jamie.e.cook@nasa.gov	NASA Goddard Space Flight Center
Daniel Glavin	Daniel.p.glavin@nasa.gov	NASA Goddard Space Flight Center
Jason Dworkin	Jason.p.dworkin@nasa.gov	NASA Goddard Space Flight Center

Jose Aponte	jose.c.aponte@nasa.gov	NASA Goddard Space Flight Center
Danielle Simkus	Danielle.n.simkus@nasa.gov	NASA Goddard Space Flight Center
Darby Dyar	mdyar@mtholyoke.edu	Mount Holyoke College/PSI
Antonio Lanzirotti	lanzirotti@uchicago.edu	University of Chicago
Stephen Sutton	sutton@cars.uchicago.edu	University of Chicago
Molly McCanta	mmccanta@utk.edu	University of Tennessee, Knoxville
Elizabeth Sklute	ecsklute@psi.edu	Planetary Science Institute
Kees Welten	kcwelten@berkeley.edu	University of California Berkeley
Kunihiko Nishizumi	kuni@berkeley.edu	University of California Berkeley
Marc Caffee	mcaffee@purdue.edu	Purdue University
Natalie Curran	natalie.m.curran@nasa.gov	Goddard Space Flight Center
Barbara Cohen	barbara.a.cohen@nasa.gov	Goddard Space Flight Center
Sarah Valencia	sarah.n.valencia@nasa.gov	University of Maryland
Catherine Corrigan	corrigan@si.edu	National Museum of Natural History
Emma Bullock	ebullock@carnegiescience.edu	Carnegie Earth and Planets Laboratory
Kate Burgess	kate.burgess@nrl.navy.mil	Naval Research Laboratory
Rhonda Stroud	stroud@nrl.navy.mil	Naval Research Laboratory
Brittany Cymes	brittany.cymes.ctr@nrl.navy.mil	Naval Research Laboratory
Jeffrey Gillis-Davis	j.gillis-davis@wustl.edu	Washington University St. Louis
Richard Walroth	rwalroth@stanford.edu	SLAC National Accelerator Laboratory
Thomas Kroll	tkroll@slac.stanford.edu	SLAC National Accelerator Laboratory
Dimosthenis Sokaras	dsokaras@slac.stanford.edu	SLAC National Accelerator Laboratory
Hope Ishii	ishii3@hawaii.edu	University of Hawaii
John Bradley	johnbrad@hawaii.edu	University of Hawaii
Roberto Colina-Ruiz	rcolina@stanford.edu	SLAC National Accelerator Laboratory
Charles Shearer	cshearer@unm.edu	University of New Mexico
Bradley Jolliff	bjolliff@wustl.edu	Washington University St. Louis
Mahesh Anand	mahesh.anand@open.ac.uk	Open University
James Carpenter	james.carpenter@esa.int	European Space Agency
Lars Borg	borg5@llnl.gov	Lawrence Livermore National Laboratory
Katherine Joy	Katherine.joy@manchester.ac.uk	University of Manchester
Lindsay Keller	lindsay.p.keller@nasa.gov	NASA Johnson Space Center
Paul Lucey	lucey@higp.hawaii.edu	University of Hawaii
Clive Neal	cneal@nd.edu	University of Notre Dame
Noah Petro	noah.e.petro@nasa.gov	NASA Goddard Space Flight Center
Harrison Schmitt	hhschmitt@earthlink.net	Harrison Schmitt Consulting
Richard Walker	rjwalker@umd.edu	University of Maryland
Alex Bradley	abradley@eps.wustl.edu	Washington University St. Louis
Adrian Brearley	brearley@unm.edu	University of New Mexico
William Cassata	cassata2@llnl.gov	Lawrence Livermore National Laboratory
Roy Cristofferson	roy.cristoffersen-1@nasa.gov	NASA Johnson Space Center
Simon Clemett	simon.j.clemett@nasa.gov	NASA Johnson Space Center
Aidan Cowley	aidan.cowley@esa.int;	European Space Agency
Catherine Dukes	cdukes@virginia.edu	University of Virginia
Kate Freeman	khf4@psu.edu	Penn State University
Amy Gaffney	gaffney1@llnl.gov	Lawrence Livermore National Laboratory
Rhian Jones	rhian.jones-2@manchester.ac.uk	University of Manchester
Randy Korotev	korotev@wustl.edu	Washington University St. Louis
Thomas Kruijer	kruijer1@llnl.gov	Lawrence Livermore National Laboratory
Gordon Love	glove@ucr.edu	University of California Riverside
Dayl Martin	dayl.martin@esa.int	European Space Agency
Matthias Maurer	Matthias.Maurer@esa.int;	European Space Agency
Alex Meshik	ameshik@physics.wustl.edu	Washington University St. Louis
Alexandre Meurisse	Alexandre.Meurisse@esa.int	European Space Agency
Richard Morris	richard.v.morris@nasa.gov	NASA Johnson Space Center
James Papike	jpapike@unm.edu	University of New Mexico

Rita Parai	parai@wustl.edu	Washington University St. Louis
Olga Pravdivtseva	olga@wustl.edu	Washington University St. Louis
Zachary Sharp	zsharp@unm.edu	University of New Mexico
Justin Simon	justin.i.simon@nasa.gov	NASA Johnson Space Center
Steven Simon	bs8@unm.edu	University of New Mexico
Corliss Kin Sio	sio2@llnl.gov	Lawrence Livermore National Laboratory
Lingzhi Sun	lzsun@higp.hawaii.edu	University of Hawaii
Romain Tartèse	romain.tartese@manchester.ac.uk	University of Manchester
Kathie Thomas-Keprta	kathie.thomas-keprta-1@nasa.gov	NASA Johnson Space Center
Michelle Thompson	thomp655@purdue.edu	Purdue University
Kun Wang	wangkun@wustl.edu	Washington University St. Louis
Josh Wimpenny	wimpenny1@llnl.gov	Lawrence Livermore National Laboratory
Michael Cato	mcato@unm.edu	University of New Mexico
Anthony Gargano	agargano@unm.edu	University of New Mexico
Mason Neuman	mdneuman@wustl.edu	Washington University St. Louis
James McFadden	mcfadde8@purdue.edu	Purdue University
Dan Moriarty	daniel.p.moriarty@nasa.gov	NASA Goddard Space Flight Center
Giulia Magnarini	Giulia.magnarini.14@ucl.ac.uk	University College, London
Patrizia Will	Patrizia.will@wustl.edu	Washington University St. Louis
Zhen Tian	t.zhen@wustl.edu	Washington University St. Louis
Chris Yen	yence@wustl.edu	Washington University St. Louis
Abbey Flom	aflom@hawaii.edu	University of Hawaii
Chiara Ferrari-Wong	cfw@hawaii.edu	University of Hawaii
Jessika Valenciano	jvalenc2@nd.edu	University of Notre Dame
Kamil Stelmach	kbs7dqw@virginia.edu	University of Virginia
Grace Minesinger	gmm9uf@virginia.edu	University of Virginia
Grant Killian	Gk3uk@virginia.edu	University of Virginia
Paul Carpenter	Paulc@levee.wustl.edu	Washington University St. Louis
Julian Rodriguez	smrodriguez@wustl.edu	Washington University St. Louis
Thomas Mitchell	tom.mitchell@ucl.ac.uk	University College, London
Peter Grindrod	p.grindrod@nhm.ac.uk	Natural History Museum London
Karen Ziegler	kziegler@unm.edu	University of New Mexico
James Dottin	jdottin@terpmail.umd.edu	Carnegie Earth & Planets Laboratory
Stu Webb	gwebb1@nd.edu	University of Notre Dame
Samantha Bell	samantha.bell@manchester.ac.uk	University of Manchester
Erick Cano	ejcano@unm.edu	University of New Mexico
Linda Ziamanesh	lsz9tp@virginia.edu	University of Virginia
Jessica Oraegbu	mfg9rv@virginia.edu	University of Virginia
Adam Woodson	akw8r@virginia.edu	University of Virginia
Jillian Maxson	jtm8hqg@virginia.edu	University of Virginia
Angelina Minocha	angelinam@wustl.edu	Washington University St. Louis
Ryan Ogliore	rogliore@wustl.edu	Washington University St. Louis
Caitlin Ahrens	Caitlin.ahrens@nasa.gov	NASA Goddard Space Flight Center
Francesca McDonald	Francesca.mcdonald@esa.int	European Space Agency
Advenit Makaya	Advenit.makaya@esa.int	European Space Agency
Nathan Bamsey	Nathan.bamsey@esa.int	European Space Agency
Thomas Rohr	Thomas.rohr@esa.int	European Space Agency
Gianluca Casarosa	Gianluca.casarosa@esa.int	European Space Agency
Matteo Appolloni	Matteo.appolloni@esa.int	European Space Agency
Robert Linder	Robert.lindner@esa.int	European Space Agency
Yuriy Butenko	Yuriy.butenko@esa.int	European Space Agency
Timon Schild	Timon.schild@esa.int	European Space Agency
Eoin Tuohy	Eoin.tuohy@esa.int	European Space Agency
Cyrille Crespi	Cyrille.crespi@esa.int	European Space Agency
Paul deMediros	Paul.demediros@esa.int	European Space Agency
Benoit Andre	Benoit.andre@esa.int	European Space Agency

Riccardo Biella	Riccardo.biella@esa.int	European Space Agency
Fiona Thiessen	Fiona.thiessen@esa.int	European Space Agency
Ryan Zeigler	ryan.a.zeigler@nasa.gov	NASA Johnson Space Center
Juliane Gross	juliane.gross@nasa.gov	NASA Johnson Space Center
Charis Krysher	charis.h.krysher@nasa.gov	NASA Johnson Space Center
Andrea Mosie	andrea.b.mosie@nasa.gov	NASA Johnson Space Center
Judith Allton	Judith.h.allton@nasa.gov	NASA Johnson Space Center
Scott Eckley	scott.a.eckley@nasa.gov	NASA Johnson Space Center
Jeremy Kent	jeremy.j.kent@nasa.gov	NASA Johnson Space Center
Julie Mitchell	julie.l.mitchell@nasa.gov	NASA Johnson Space Center
Cecilia Amick	cecilia.l.amick@nasa.gov	NASA Johnson Space Center
Ernest Lewis	ernest.k.lewis@nasa.gov	NASA Johnson Space Center
Romy Hanna	romy@jsg.utexas.edu	University of Texas, Austin
Richard Ketcham	ketcham@jsg.utexas.edu	University of Texas, Austin
David Edey	dave.edey@utexas.edu	University of Texas, Austin
Evan O'Neal	evan.w.o'neal@nasa.gov	NASA Johnson Space Center
Francis McCubbin	francis.m.mccubbin@nasa.gov	NASA Johnson Space Center
Tabb Prissel	tabb.c.prissel@nasa.gov	NASA Johnson Space Center

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