3.5 THERMOLUMINESCENCE DOSIMETRY

Contact Person(s) : Gladys Klemic

3.5.1 SCOPE

Research at EML is directed toward advancements in environmental applications of thermoluminescence dosimetry (TLD). This includes investigations of problems associated with low-level measurements of the gamma- and cosmic-ray natural background (de Planque and Gulbin, 1985; Julius and de Planque, 1984; Gulbin and de Planque, 1983, 1984), as well as techniques for the separation of neutron and gamma components from artificial sources (Klemic et al., 1996). EML organizes the International Intercomparisons of Environmental Dosimeters, a voluntary program of testing and research on integrating dosimeters for the measurement of environmental radiation (Klemic et al., 1995; Maiello et al., 1990a,b, 1995; de Planque and Gesell, 1986) that attracts participants from around the world.

Consistent procedures for the preparation, packaging, recordkeeping and readout, along with many QC checks, are necessary to obtain reliable results. The procedures described here have been shown to result in high quality measurements that are suitable for low-level environmental radiation monitoring and for TLD research purposes (see also American National Standards Institute Report, ANSI N545-1975, and the future release of ANSI N13.37, which will replace it).

3.5.2 SPECIAL APPARATUS

1. TLD phosphors:

Lithium fluoride dosimeters - ^{7}LiF:Mg,Ti chips are used for most applications. They are 3.2 x 3.2 x 0.89 mm, 24 mg chips manufactured by Harshaw/Bicron Co., 6801 Cochran Rd., Solon, OH 44139-3395 (TLD-700).

Aluminum oxide dosimeters - Al_2O_3:C chips are presently used only for research purposes. They are 5 mm in diameter and 1 mm thick and are manufactured by Victoreen, Inc., 6000 Cochran Rd., Solon, OH 44139-3395 (Model 2600-80).

Calcium fluoride chips and bulb dosimeters - EML's historical experience has included bulb-type dosimeters and other types of chips such as CaF_2:Mg (Gulbin and de Planque, 1983; Gulbin and de Planque, 1984); however, these phosphors are not presently in use and will not be covered here.

2. Annealing equipment:

*High temperature furnace** - A furnace with constant temperature capability up to 1000ºC (manufactured by Blue M Electric Co., Blue Island, IL 60406) is used for high temperature annealing.

Low temperature furnace - This furnace is maintained at 100ºC for low temperature annealing (manufactured by Fisher Isotemp, Pittsburgh, PA 15219).

Planchet - An EML-designed, platinum-plated copper planchet is used for chip annealings. The tray is indented so that chips may be identified by placement (see Figure 3.9).

Brass heat sink - A brass heat sink, 5 x 5 x 21 cm, is mounted on an aluminum base that is placed on steel unistrut bars to allow uniform cooling of chips (see Figure 3.9).

3. Controlled lighting:

Gold fluorescent lights with no ultraviolet emissions.

Dark room shades.

Incandescent bulbs, including a 25-W red bulb for minimal lighting.

4. ^{137}Cs source:

A 2 Ci collimated NIST traceable ^{137}Cs source is used for calibration. Usual exposure distance is 2 m, where the beam is uniform within a radius of at least 3.0 cm.

**A new controlled atmoshpere oven (manufactured by GS Lindberg/Blue M., Watertown, WI) has recently been installed for research but it is not yet in regular use.*

5. TLD readers:

The TLD reader room is air-conditioned to maintain a constant temperature (~ 20ºC) year-round. Three TLD readers are presently in use:

EML reader - This reader was built by EML and has been in use since 1971. It uses linear pan heating and measures the TL signal with a high-gain, low-noise photo-multiplier tube cooled to 15ºC below room temperature. An internal light source is used to verify the electronic stability of the system. Power is applied to a heating element silver-soldered to a thin silver heater pan. The chip is positioned manually and centered in the pan by a removable platinum-plated, stainless-steel disk with a central hole for the TLD. This disk suppresses the infrared signal from the heater pan and provides a reproducible geometry for chip placement. Heating parameters are adjustable up to a maximum temperature of about 550ºC. Purified nitrogen gas flows through the heating chamber at a rate of about 2.4 L min^{-1} to suppress any nonradiation induced TL signal. A strip chart records the glow curve and temperature profile with a selected glow peak region of interest indicated by pen offsets. The integral "counts" in this region is indicated on a numerical display.

Victoreen Model 2800M - A commercially available reader with many of the same features as the EML reader using updated technology, it will eventually replace the aging EML reader. Readout data can be sent to a printer or personal computer.

Automatic hot gas reader (TNO) - Built by Radiologische Dienst TNO at the Netherlands Organization for Applied Research, this reader is different from the other two, both in heating methods and chip handling. It uses three jets of preheated nitrogen gas (about 190ºC constant temperature, not a linear profile) for reproducibility and speed. Chips are handled automatically at a rate of 150 per hour up to 1000 chip readings. Readout parameters and output are controlled and stored by a personal computer. This reader is especially suitable for large-scale studies and routine monitoring.

6. Lead shield:

A 10-cm thick lead shield is used to store TLDs after preparation or before readout.

3.5.3 PREDEPLOYMENT PREPARATION OF CHIPS

A. Batch preselection.

Before chips are to be used for measurements, a set (typically 200 to 300 chips) from a single manufacturing batch is tested for uniformity as follows:

1. Clean and anneal chips as described in Section B.

2. Give chips a single exposure to ^{137}Cs (typically on the order of 70 µGy in air, or 8 mR).

3. Read out the chips (see Table 1 for readout parameters).

4. Assign chips with readings that agree to within 30% of each other to the same group. Outliers should be flagged and removed from the group. (Note: A tighter criterion of 5-10% may be used for special applications.)

5. Assign each chip a unique identification number to be permanently retained.

B. Cleaning/annealing.

1. Manually clean individual chips before each use with methanol and a cotton swab, handling chips with forceps or a vacuum pen under appropriate lighting conditions (see Table 3.2) (Freeswick and Shambon, 1970).

2. Anneal chips as follows:
   LiF: 1 h at 400ºC, 1-min cool-down on heat sink, followed by 2 h at 100ºC.
   Al_2O_3: 10 min at 400ºC.

3. Cool chips to room temperature on heat sink.

4. Store chips in the lead shield if they are not going to be used immediately. Keep careful records of the date and time that the chips are placed in the lead shield, as well as when they are removed for deployment.

C. Dosimeter packaging.

Packaging should be light-tight, moisture proof, and thick enough to provide electronic equilibrium and to shield against environmental beta radiation. The EML dosimeter consists of a commercially available black lucite box in which additional black lucite pieces have been bonded in place to provide depressions for holding the individual chips (see Figure 3.10). It has outside dimensions of 2.9 x 2.9 x 0.9 cm. Each chip is surrounded by lucite with a minimum thickness of 2.8 mm (about 320 mg cm^{-2}). A dosimeter usually consists of five chips, though the container can hold up to 10 chips. To ensure water-tightness, the lucite box is placed inside a thin plastic bag which is heat sealed or secured with fiber tape.

3.5.4 ENVIRONMENTAL DEPLOYMENT

Environmental TLDs are usually deployed for 1-3 months. A preselected uniform batch (see Section 3.5.3.A) with enough TLDs to cover at least three monitoring cycles is maintained exclusively for use in environmental monitoring (i.e., while one set is in the field, there are enough dosimeters for two replacement sets). A set of environmental dosimeters for a monitoring cycle includes six control dosimeters prepared with the field dosimeters, as described in Section 3.5.3.B. All dosimeters are stored in the lead shield after preparation until they can be deployed, noting date and time. Two of the controls will be used to determine the exposure rate in this shield (STORAGE CONTROLS) and are thus stored there for the duration of the field cycle. The other four are used for calibration of the system (CALIBRATION CONTROLS) and will be discussed in Section 3.5.5.D.

If the field site is far from the laboratory, it is necessary to use additional controls to account for exposure received in transit (TRANSIT CONTROLS). These controls must be kept with the field dosimeters at all times except during field deployment, when they must be kept somewhere where the exposure rate is known or can be measured independently.

The dosimeter is hung 1 m above the ground, away from large structures that may provide shielding or additional exposure (see Figure 3.11). Usually the dosimeter is left hanging freely to rotate in the wind for isotropic angular exposure. At the end of the field cycle, the dosimeters are returned to the lead shield until they are read out, and a replacement set is deployed in the field.

3.5.5 READOUT AND CALIBRATION

A. Reader checkout.

1. Prepare the reader for use by setting the appropriate reader parameters as shown in Table 3.2.

2. Check the reader's dark current, the heating chamber current (background of empty chamber), and the response of the reader to the internal light source.

3. Read out a test chip several times to check for anomalous results.

B. Preread annealing.

1. Remove the FIELD dosimeters from the lead shield, noting the date and time.

2. Perform a preread anneal for LiF chips at 100°C for 10 min. (No preread anneal is used for Al_2O_3 chips.)

C. Initial readout.

1. Read out **one** chip from each FIELD dosimeter. After it returns to room temperature, the chip is read a second time to measure the background of the system.

2. Temporarily discontinue readout at this point to prepare the calibration dosimeters.

D. Calibration.

Four dosimeters are used to calibrate the system during readout. Two of these are from the set originally prepared at the start of the field cycle, and two are taken from the set of replacement dosimeters just prepared. Thus, while each prepared set includes four calibration dosimeters, two will be used right away to calibrate the returning field set and the other two are stored in the lead shield for the duration of the field cycle to be used at the following readout. The calibration is performed as follows:

1. Examine the range of reader net counts from the initial readout of each field dosimeters to determine the calibration exposures to use. Choose three calibration exposures to bracket the readings given by the field dosimeters and to provide a

check on the linearity of the system. (The approximate counts per unit exposure is known from previous readouts.)

2. Remove the CALIBRATION CONTROLS from the lead shield, noting the date and time of retrieval.

3. Give one CALIBRATION CONTROL an exposure that is expected to yield net counts in the range of the lowest result found for the FIELD dosimeters. Another CALIBRATION CONTROL is given an exposure corresponding to the highest reading, and the other two receive an exposure in the middle of this range.

E. Resuming readout.

1. Remove the STORAGE CONTROL dosimeters from the lead shield, noting the date and time.

2. Anneal all of the control dosimeters in the case of LiF TLDs (the four CALIBRATION CONTROLS as well as the two STORAGE CONTROLS) for 10 min at 100ºC.

3. Intersperse the STORAGE CONTROL and CALIBRATION CONTROL dosimeters among the FIELD dosimeters. Read out one chip from each dosimeter before going to the next chip in any dosimeter. (This provides a QC check against any variations in the system during readout.)

3.5.6 ANALYSIS OF RESULTS

An interactive program that runs on a personal computer (written in Fortran) handles the data analysis. A sample input file is shown in Appendix A. It includes for each dosimeter: date and times of preparation, deployment, return, and readout; gross counts; background counts; and time of exposure to the cesium source. The program determines the field exposure by correcting for exposure received in storage as measured by the storage controls and then converting counts to exposure by using the calibration controls. The user is given the option of rejecting outliers, and the statistical error is propagated throughout each step in the calculation. The program returns the net field exposure and exposure rate and the field deployment time for each field dosimeter. It also gives information about the exposure rate in storage, the calibration factor, and any rejected readings. A sample output file is shown in Appendix A. Details of the calculations are given below.

A. Computing net counts and standard error.

The mean net counts for each dosimeter is:

$$X = \sum_{i=1}^{n} (x_i) \div n$$

where

x_i = gross counts - background counts of *i*th chip.
n = number of chips per dosimeter (n = 5 usually).

The standard error of the mean for each dosimeter is:

$$err_x = \frac{\sigma}{\sqrt{n}} = \sqrt{\frac{\sum_{i-1}^{n} (x_i - X)^2}{n(n-1)}}$$

B. Outlier check.

The program checks for outliers among the individual chip readings in a dosimeter. Two different criteria are used:

1. *"Extreme Values"* - Individual chip net counts that are > 150% or < 50% of the dosimeter average **X** are flagged. This test would find missing chips or noise spikes.

2. *"r Test"* - This is an outlier test for gaussian distributions (Proschan, 1969). The net counts for the five chips are ranked in order of smallest to the largest, such as x_1, x_2, x_3, x_4, x_5. If the ratio r = $(x_2 - x_1) \div (x_5 - x_1)$ is larger than 0.780, chip x_1 is flagged. Similarly if the ratio r = $(x_5 - x_4) \div (x_5 - x_1)$ is larger than 0.780, chip x_5 is flagged. This is a more sensitive test that can identify outliers that would be missed by the extreme value test. While it is redundant for values beyond 150% of the mean, it

could miss the very low extreme values so both tests are needed. (Proschan describes more sensitive tests to apply when more than 7 chips are used.)

In either criterion, the user is then given the option of omitting flagged chips from the analysis. No chips are automatically rejected: the final decision is made by the user. (In practice data are rarely rejected, and then usually for obvious reasons.) If a chip is rejected, the mean net counts and standard error are recalculated for that dosimeter, and a note appears in the output file.

C. Storage exposure rate correction factor.

$$S = \left(\tfrac{1}{2}\right)\left(X_{SC1} + X_{SC2}\right) \div t_{SC}$$

where

S = storage correction factor,
X_{SC1} = mean net counts for STORAGE CONTROL #1,
X_{SC2} = mean net counts for STORAGE CONTROL #2, and
t_{SC} = time storage controls were in lead shield *(calculated by subroutine from dates in input file)*

The standard error is propagated through this calculation as:

$$err_S = \frac{1}{2t_{SC}}\sqrt{\left(err_{SC1}^2 + err_{SC2}^2\right)}$$

With err_{SC1} and err_{SC2} being the standard error of the storage controls as defined in Section A above. (The error associated with the storage time is negligible, estimated at < 0.01%.)

D. Calibration factor.

$$C = \left(\frac{1}{4}\right)\sum_{i=1}^{4}\left(X_{CCi} - St_{CCi}\right) \div exposure_{CCi}$$

where

C = calibration factor,
X_{CCi} = mean net counts of CALIBRATION CONTROL #*i*

t_{CCi} = time *i*th CALIBRATION CONTROL was kept in lead shield *(calculated by subroutine from dates in input file)*,

$exposure_{CCi}$ = cesium exposure given to *i*th CALIBRATION CONTROL *(calculated from decay corrected known source strength and time of exposure)*.

The standard error propagated through this calculation is then:

$$\mathrm{err_C} = \frac{1}{4}\sqrt{\sum_{i=1}^{4}\left(\frac{\mathrm{err}_{CCi}^2 + err_S^2\, t_{CCi}^2}{\mathrm{exposure}_{CCi}^2}\right)}$$

(Uncertainties in cesium calibration exposure are treated as systematic rather than statistical errors and are treated separately.)

The program also performs a linear regression on the corrected counts vs. exposure for the four calibration dosimeters. The goodness of fit is a check on the linearity of the system and the slope may be compared to **C**.

E. Field exposure.

The field site exposure (f) is a function of the quantities calculated above. The algorithm used by the program to calculate field site exposure may be summarized as:

$$F = f(X_F, t_F, S, C) = \frac{(X_F - S\,t_F)}{C}$$

where

X_F = mean FIELD dosimeter counts

t_F = time FIELD dosimeters were kept in a lead shield *(calculated by subroutine from dates in input file)*,

F. Error analysis.

Total uncertainty = statistical error at 95% confidence + estimated systematic error

While the standard error was propagated through all the above calculations using the analytical propagation of error formulas, for the last step it is easier to calculate the statistical error numerically as shown below (Bevington and Robinson, 1992).

$$\text{Statistical err} = 2.776\sqrt{\left(a^2 + b^2 + c^2\right)}$$

where

2.776 = 95% confidence interval for 5 chips
(4 degrees of freedom)

$$a = \frac{\left(\left[X_F + err_F\right] - St_F\right)}{C} - \frac{\left(X_F - St_F\right)}{C}$$

$$b = \frac{\left(X_F - \left(S + err_F\right)t_F\right)}{C} - \frac{\left(X_F - St_F\right)}{C}$$

$$c = \frac{\left(X_F - St_F\right)}{C + err_C} - \frac{\left(X_F - St_F\right)}{C}$$

Systematic error is estimated on a case-by-case basis and added linearly to the statistical error for reporting the final results. The estimated systematic uncertainty associated with EML's cesium source is 2.5%.

G. Situations involving transit controls.

In cases where TRANSIT CONTROLS are required, the field exposure (Section E) calculation would instead be:

$$F = \left(X_F - T\right)/C$$

where

T = mean TRANSIT dosimeter counts corrected for exposure received in storage.

If it happens that the TRANSIT CONTROLS are stored in the same lead shield as the other storage controls during the field cycle (time = t_F), then

$$T = \left(X_T - St_F\right)$$

where

X_T = mean TRANSIT dosimeter counts.

More likely the TRANSIT CONTROLS will be stored in a different lead shield near the field site, in which case the storage exposure must be measured by some other means.

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TABLE 3.2

SUMMARY OF TLD PARAMETERS USED

ANNEALING		
	Predeployment	Prereadout
LiF	400ºC 1 h 100ºC 2 h	100ºC 10 min
Al_2O_3	400ºC 10 min	none

LIGHTING CONDITIONS	
LiF	no UV: gold fluorescent or incandescent
Al_2O_3	darkroom

READ OUT Linear Heating: Victoreen Reader				
	Starting Temp	Heating Rate	Heating Time	Integration Period*
LiF	100ºC	10ºC sec^{-1}	30 sec	~ 10 - 20 sec
Al_2O_3	100ºC	10ºC sec^{-1}	20 sec	~ 5 - 20 sec

Table 3.2 (Cont'd)

READ OUT Linear Heating: EML			
	Heating Rate	Maximum Temperature	Integration Period* (adjusted visually)
LiF	10ºC sec^{-1}	~ 330ºC	peaks 3,4 &5
Al_2O_3	10ºC sec^{-1}	~ 300ºC	whole peak

READ OUT Constant Temperature Hot Gas: TNO		
	Heating Time	Integration Period*
LiF	11 sec	1.3 - 10.0 sec
Al_2O_3	13 sec	1.7 - 12.0 sec

*Integration period adjusted as needed to include entire peak.

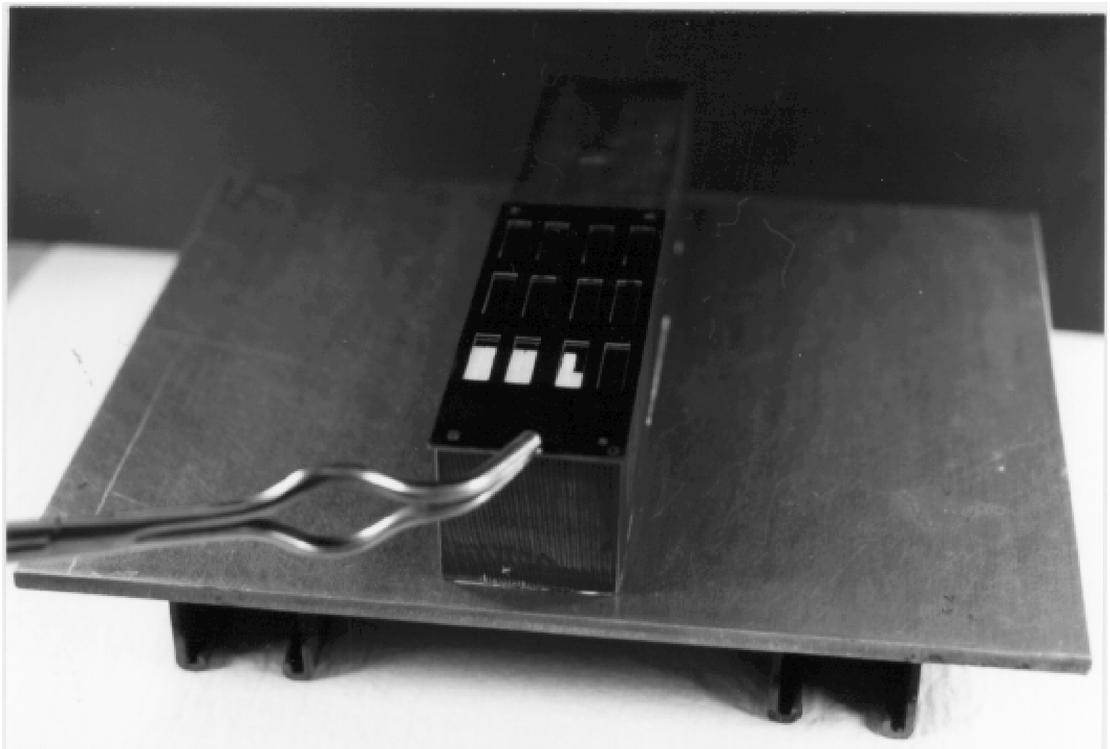

Figure 3.9. EML-designed planchet used for chip annealings, shown on brass heat sink. Chip identity is retained by position in planchet.

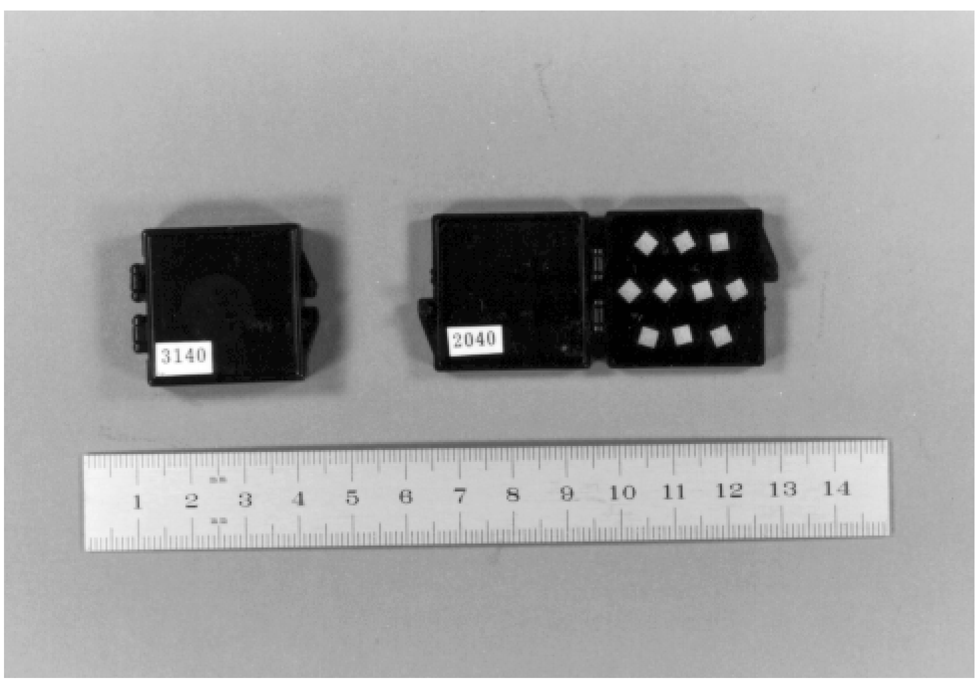

Figure 3.10. EML's light-tight lucite dosimeter package (320 mg cm^{-2}), shown

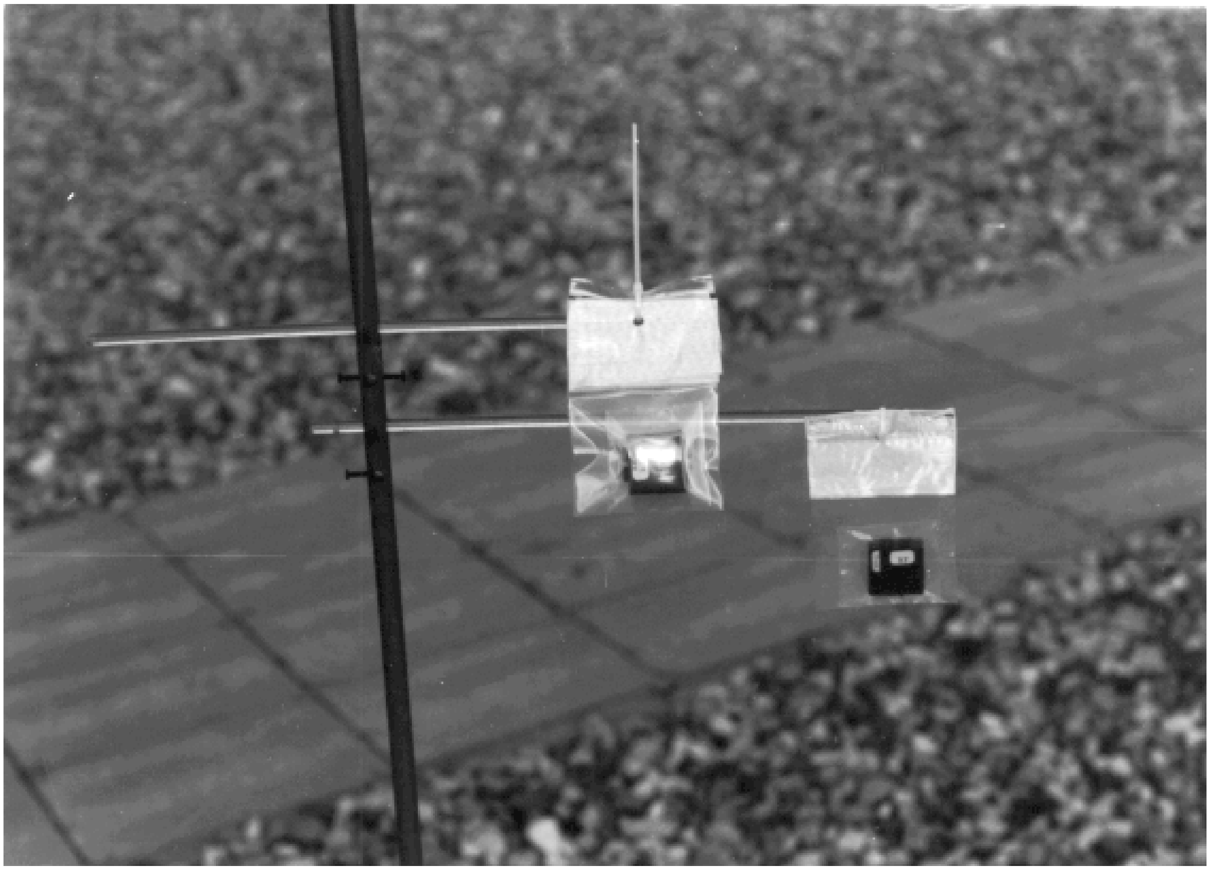

Figure 3.11. EML dosimeter deployed at field site. Lucite box is sealed in plastic bag to protect from moisture, and hung ~ 1 m above ground.

APPENDIX A

SAMPLE INPUT FILE   Environmental TLD Measurements

SITE	ID	INTO LEAD SHIELD	DEPLOYED	RET. LEAD SHIELD	READ OUT	Counts GROSS	Counts BKD	Cs Expos min
Shrm	3140	02/21/93	02/26/93	04/27/93	10/20/93	430	36	0.00
		19:23	15:55	07:37	09:45	425	32	
						417	34	
						414	30	
						447	38	
Roof	3100	02/21/93	03/01/93	05/25/93	10/20/93	534	35	0.00
		19:23	15:20	12:45	09:45	507	33	
						556	28	
						536	36	
						550	36	
Chstr	3110	02/21/93	02/23/93	05/19/93	10/20/93	808	29	0.00
		19:23	14:45	12:00	09:45	812	36	
						806	29	
						803	30	
						783	34	
Chstr	3120	02/21/93	02/23/93	05/19/93	10/20/93	811	30	0.00
		19:23	14:45	12:00	09:45	802	30	
						764	34	
						739	38	
						764	33	
Sphr	3130	02/21/93	03/01/93	05/25/93	10/20/93	319	35	0.00
		19:23	15:20	12:45	09:45	349	31	
						333	30	
						338	30	
						347	31	
Stor	3200	02/21/93	00/00/00	00/00/00	10/20/93	265	29	0.00
Ctl		19:23	00:00	00:00	09:45	257	32	
						264	27	
						267	32	
						253	29	
Stor	3190	02/21/93	00/00/00	00/00/00	10/20/93	241	32	0.00
Ctl		19:23	00:00	00:00	09:45	269	36	
						329	32	
						244	32	
						239	32	
Calib 1	3150	02/21/93	00/00/00	00/00/00	10/20/93	478	31	3.50
		19:23	00:00	00:00	09:45	511	32	
						471	35	
						489	39	
						486	36	
Calib 2	3160	02/21/93	00/00/00	00/00/00	10/20/93	656	31	6.0
		19:23	00:00	00:00	09:45	643	30	
						675	34	
						618	41	
						643	34	
Calib 3	3000	04/23/93	00/00/00	00/00/00	10/20/93	614	30	6.0
		13:00	00:00	00:00	09:45	598	34	
						610	32	
						666	38	
						597	30	
Calib 4	3050	04/23/93	00/00/00	00/00/00	10/20/93	771	28	9.0
		13:00	00:00	00:00	09:45	798	31	
						791	32	
						777	32	
						796	29	

APPENDIX A (Cont'd)

SAMPLE OUTPUT FILE

Environmental TLD Measurements (with statistical uncertainty at 1 σ)

SITE	DEPLOYMENT		NETmR	EXPOSURE RATE μR/h	FIELD h
Shrm	2-26-93	4-27-93	9.12	6.37 ± .25 (3.9%)	1431.70
Roof	3-01-93	5-25-93	14.53	7.13 ± .22 (3.1%)	2037.42
Chstr	2-23-93	5-19-93	24.68	12.11 ± .29 (2.4%)	2037.25
Sphr	3- 1-93	5-25-93	6.73	3.30 ± .17 (5.2%)	2037.42

Stor. Corr. Factor: .0448 ± .00150 cts/h (3.4%)

Ave. Calibration Factor: 25.028 ± .428 cts/mR (1.7%)

Lead Shield Exp. Rate: 1.789 ± .067 micro-R/h (3.8%)

SITE	SN	Net Cts	Store h	Cor. Cts	err	% err	sd	%sd
Shrm	3140	422.60	4342.67	228.19	± 8.0	(1.1%)	10.5	4.58
Roof	3100	531.00	3736.95	363.71	± 9.5	(1.5%)	17.3	4.75
Chstr	3110	784.90	3737.12	617.60	± 10.0	(1.1%)	26.3	4.26
Sphr	3130	335.80	3736.95	168.51	± 8.3	(1.8%)	13.6	8.08
Stor Ctl	3200	255.40	5774.34	255.40	± 3.4	(1.3%)	7.5	2.94
Stor Ctl	3190	261.60	5774.34	261.60	± 17.0	(6.5%)	38.0	14.53
Calib 1	3150	482.40	5774.34	223.90	± 11.2	(1.5%)	15.9	7.12
Calib 2	3160	645.00	5774.34	386.50	± 12.5	(1.4%)	20.0	5.17
Calib 3	3000	614.20	4316.72	420.95	± 13.2	(1.9%)	25.8	6.13
Calib 4	3050	782.20	4316.72	588.95	± 8.8	(0.8%)	13.3	2.26

APPENDIX A (Cont'd)

SAMPLE OUTPUT FILE continued

..........CALIBRATION INFORMATION..........

Name	SN	Expos mR	cts/mR	err	%err
Calib 1	3150	9.229	24.260	± 1.216	(5.0%)
Calib 2	3160	15.821	24.429	± 0.787	(3.2%)
Calib 3	3000	15.821	26.607	± 0.836	(3.1%)
Calib 4	3050	23.732	24.817	± 0.371	(1.5%)

Fit to line:

slope = 25.084 (cts/mR)
y intcpt = -0.05930
rnorm = 28.031
ierr = 0

The following packets had outlier data rejected:

(none)