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Etching Procedure Optimization: Summer Project 2015

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Prepared by:

Evan Knouse 2015 Summer Student National Laboratories	________________________
Date:	________________________

Reviewed and Approved by:

Jason Sadowski, MSc., P.Eng Laboratory Manager National Laboratory	________________________
Date:	________________________

2 Revision History

Revisions to this document should be made following procedure *WP-15, Document and Record Control* and submitted to the appropriate person for review.

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Sample calculation of dilution:

$M_1V_1 = M_2V_2$

(2.5 M) V_1=(2.0 M)(1.0 L)

$V_1 = 0.8\ L$

Add 200 ml water to 800 ml 2.5 M NaOH solution to get 1.0 L of 2.0 M NaOH solution.

Sample calculation of exposure time, t:

$$t = \frac{1005}{A * \varepsilon_G}$$

$$t = \frac{1005}{1022.3Bq * 0.001037}$$

t =948.0013255s

$t = 948s \pm 5s$

Sample calculation of sample size, n:

$$n = \left(\frac{z_{\alpha/2}\sigma}{E}\right)^2$$

$z_{\alpha/2} = 1.96\ (for\ 95\%\ confidence)$, $\sigma = 0.071562$, E= 0.05

$$n = \left(\frac{1.96 * 0.071562}{0.05}\right)^2$$

$$n = 7.869317597\ \approx 8$$

<u>Sample calculation of sample variance, s^2:</u>

$$s^2 = \frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}$$

*Using data from Table 7

$$s^2 = \frac{18608.4064}{11}$$

$$s^2 = 1691.6733$$

<u>Sample calculation of pooled variance estimator, s_p^2:</u>

$$s_p^2 = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}$$

*Using data from Tables 7 & 8

$$s_p^2 = \frac{95 * 15045.56 + 11 * 1691.6733}{96 + 12 - 2}$$

$$s_p^2 = 13659.7793$$

5. Introduction

The purpose of the experiment is to determine the optimal etching procedure for chemically etching Kodak LR115 type II films currently used by the RSIC. To do so, factors including etching time, temperature, molarity, and PH will be tested for their effects on the quality of the films.

6. Background

The detection of Uranium-238 decay progeny such as Radon-222, an inert, radioactive gas, as well as long-lived radioactive dusts, is central to the safety of those working on uranium mining operations. It is therefore imperative that action is taken to ensure the accurate measurement of the exposure of personnel to these radioactive sources. With the advent of solid-state nuclear track detectors, such as Kodak LR115 films, a feasible dosimetry method for miners became available. Using equipment designed by both French and Canadian scientists, the RSIC operates a dosimetry service for many of the mining operations in Saskatchewan. The equipment makes use of the Kodak LR115 films by drawing air into a small plastic chamber, containing collimators, which discriminate between various Uranium-238 progenies, such as Radon-222. The collimators are covered at one end by the LR115 film, which acts as a screen to be exposed to alpha particles emitted by the radionuclide that enters the dosimeter head. Upon exposure, the films will be left with small tracks in them, caused by the alpha particles emitted during certain decays. The number of tracks detected is proportional to the amount of radioactivity a miner is exposed to, and the RSIC analyzes the films. Since the alpha tracks are not visible to the naked eye, a special procedure must be performed to magnify the tracks. The process required involves chemically etching away the emulsion layer of the LR115 film, which includes heating a bath of sodium hydroxide solution, to a specific temperature, and placing the exposed films into the solution, for a specified period of time. Under the correct conditions, the alpha tracks will be magnified, and a technician, using a microscope with digital imaging capabilities, can then count them. On the advisement of technical documents provided by DOSIRAD in France, the RSIC currently operates the chemical etching process with specific parameters of 95 minutes for time, 60°C for temperature, 2.5 M NaOH etching solution, and a rinse in tap water (~ PH 7) over night. However, the optimal etching parameters are unknown, and are the motivation for the experiment at hand. This is because of the effect on the overall quality, reliability and accuracy of the dosimetry service provided by the RSIC that the etching process has. A chemical etching process, operating at the optimal settings, would ensure the RSIC provides only the highest quality service to the many workers and families who require radiation safety. Another motivator for the experiment is the anomalous background counts being observed by the RSIC. Since all operations are done under virtually identical environmental conditions, the background is expected to remain essentially constant between analyses. However, The RSIC has noticed an unpredictable pattern in the background counting data. For these reasons, an investigation into the effects of etching parameters on film registration efficiency, and background counts, is necessary.

7. Materials and Methods

The equipment used for the experiment was as follows:

- RSIC dosimeter head
- Kodak LR115 type II films
- 1025 Becquerel Americium-241 test source (K9-744, Jan.1st, 2014)
- 670 Becquerel Americium-241 test source (H6-989, Feb.1st, 2011)
- 3030 Becquerel Americium-241 test source (FP-227, Mar.7th, 1997)
- 2.0,2.5,3.0 M NaOH etching solution
- Julabo TW20 heat bath
- 1 M Hydrochloric acid (HCl)
- 1L Erlenmeyer flask
- Hydrometer
- PH test probe
- Digital scale
- Metallic basins (for etching, rinsing)
- Stopwatch
- Microscope
- Imaging software

To begin with, the sample size required for the experiment needs to be determined. To do so, the following equation should be used:

$$n = \left(\frac{z_{\alpha/2}\sigma}{E}\right)^2 \qquad (1)$$

, where $z_{\alpha/2}$ = critical value (obtained from z-score table)
σ = population standard deviation
E = margin of error

Note that of particular importance to this type of experiment is the time required for exposures and etches, so special attention should be paid to the sample size calculated here. If the sample size is too large to be feasible or too small to be accurate, adjustments can be made to the allowed margin of error, if necessary. The standard deviation can be estimated from previous etch checking data, or otherwise. This quantity may also be adjusted slightly to deliver the correct sample size.

With the sample size calculated, film pieces must be cut, scribed, and exposed. The number of films necessary will depend on the size of the factorial-designed experiment, the number of samples required, and multiplied by two to account for exposed films and background films. For example, running a two-level, four-factor factorial experiment requires 2^4=16 “runs”, each requiring eight samples of both exposed and background films for a total film count of 16x8x2=256 films. The ideal exposure regime should be determined prior to beginning the experiment. The exposure plan should take into consideration the time needed per exposure, the film registration efficiency achievable given a certain apparatus, the count of alpha particle tracks expected based on the source, and the consistency of the exposure measurements. In order to make the registration efficiency calculations, the following equation should be used:

$$\varepsilon_R = \frac{\#\alpha\ measured}{\#\alpha\ expected} = \frac{N_{counts} * \kappa_{IAS}}{A * t * \varepsilon_G} * 100 \qquad (2)$$

, where N_{counts} = average number of alpha tracks, from imaging software

A = activity of source, corrected for current date (see Analysis)

ε_G = Geometric efficiency of exposure apparatus (see Appendix)

t = length of time for exposure

κ_{IAS}= Image acquisition system scaling factor

Next, the calculations needed to adjust the molarity of the NaOH solution will need to be made. To adjust the molarity of the NaOH solution, the volume of base solution required, whether it is water or otherwise, will need to be estimated. Use an Erlenmeyer flask to mix the solutions. Since the molar mass of NaOH is approximately 39.99711g/mol, adding 40 grams of NaOH (possibly lye) to 1 L of any molarity of NaOH solution will increase the molarity by 1 M. For example, adding 40 grams of NaOH to 1 L of 2.0M NaOH solution will increase the solution to 3.0M. To dilute a solution down to a lower molarity from a higher one, use the dilution equation (see Appendix). Placing a hydrometer into a large cylinder filled with solution can test the molarity of the solution. By measuring the specific gravity of the solution, the density, and therefore the concentration of the mixture, can be measured. The hydrometer should read 1.04 if the solution of NaOH is at 1.0 M. For higher molarities, simply add .02 for every half molarity. For example, a 3.0 M solution would read 1.12 on the hydrometer.

Next, the PH of the rinse water should be adjusted. This step depends on the current PH of the water that will be used for rinsing. The PH most used for rinse water is 3. The calculation for precisely how much hydrochloric acid that will be necessary to reach PH 3 is not straightforward, and a trial-and-error approach will be more appropriate in most cases. Using a small volume syringe, on the order of hundreds of microliters, drop consistent amounts of hydrochloric acid into the rinse water, making sure to allow time for the PH probe to reach equilibrium. Stirring may be done to speed up the equilibrium process. In general, if tap water is used, it is slightly alkaline, and approximately 3.0-5.0 ml of HCl will be required to get to a PH of 3. Make sure to continually wait for relative equilibrium before adding more hydrochloric acid.

Once all of the necessary mixtures have been made, and the required films have been exposed and labelled, the etching process can begin. To start, the solution of NaOH can be poured into one of the etching basins. The heating bath can be filled with water, but not so much as to overflow into the NaOH solution basin. Surrounding the etching basin in water allows for a more consistent heating environment. Next, place the solution basin into the water in the heating bath, and turn the bath on. On the apparatus, set the temperature to the desired level, and wait for the bath to heat up. If it has not already been done, fill an appropriately sized rinsing bath with water and adjust the PH to 3 with hydrochloric acid. Then, fill a large rinsing bath with tap water for an over-night rinse. Once the bath has reached the necessary temperature, place the racks of films into the now heated etching solution basin as quickly as possible, and begin a timer. When the etching time has concluded, remove the racks of films and place in the first, acidified rinse. Rinse for approximately 30-45 minutes in this bath, then remove and place into the tap water rinse bath. Leave the film racks there over-night. The next day, remove the film racks from the rinse and place on a mat, or paper towels, and leave to dry.

After the films have had a reasonable amount of time to dry, they may be removed from the racks and placed onto glass slides for analysis. Using a microscope and imaging acquisition software, record the counts of alpha particle tracks from the exposed films and the background films. This data will be used in the calculations of registration efficiency, background counts, and signal/background ratio.

In order to make significant comparisons between the data collected and previous etch check and background data, the following equation should be used:

$$t_0 = \frac{\overline{x_1} - \overline{x_2} - \Delta_0}{s_p\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \qquad (3)$$

, where $\bar{x}_1 = sample\ 1\ mean,$

$\overline{x_2} = sample\ 2\ mean,$

$\Delta_0 = target\ for\ comparison,\ s_p^2 =$

$pooled\ estimator\ of\ population\ variance,$

$n_1 = sample\ 1\ size$

$n_2 = sample\ 2\ size$

8. Experiment

For the initial screening experiment, the factor levels chosen were as follows:

- A – Time → High - 105 minutes
  Low - 60 minutes
- B - Temp.→ High - 65°C
  Low - 50°C
- C – Molarity → High – 3.0 M
  Low – 2.0 M
- D – PH → High – 3
  Low – 7
- Films requiring exposure were exposed with Am-241 source K9-744 for 15.8 minutes, using the CAIRS dosimeter head apparatus.

<u>Experiment 1- #1</u>

- Time – Low – 60 minutes
- Temp. – Low - 50°C
- Molarity – Low – 2.0 M
- PH – Low – 7

Films used:
EX01F15-EX08F15
BG01F15-BG08F15
Date: June 29th, 2015
Etching start time: 11:05 am
Etching finish time: 12:06 pm
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1- #2</u>

- Time – High – 105 minutes
- Temp. – Low - 50°C
- Molarity – Low – 2.0 M
- PH – Low – 7

Films used:
Date: June 29th, 2015
EX09F15-EX016F15
BG09F15-BG16F15
Etching start time: 1:55 pm
Etching finish time: 3:35 pm
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#3</u>

- Time – Low – 60 minutes
- Temp. – High - 65°C

- Molarity – Low – 2.0 M
- PH – Low – 7

Films used:
EX17F15-EX24F15
BG17F15-BG24F15
Date: June 30th, 2015
Etching start time: 9:56 am
Etching finish time: 10:57 am
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#4</u>

- Time – High – 105 minutes
- Temp. – High - 65°C
- Molarity – Low – 2.0 M
- PH – Low – 7

Films used:
EX25F15-EX32F15
BG25F15-BG32F15
Date: June 30th, 2015
Etching start time: 12:08 pm
Etching finish time: 1:54 pm
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#5</u>

- Time – Low – 60 minutes
- Temp. – Low - 50°C
- Molarity – Low – 2.0 M
- PH – High – 3

Films used:
EX33F15-EX40F15
BG33F15-BG40F15
Date: June 30th, 2015
Etching start time: 2:40 pm
Etching finish time: 3:40 pm
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#6</u>

- Time – Low – 60 minutes
- Temp. – Low - 50°C
- Molarity – High – 3.0 M
- PH – Low – 7

Films used:

EX41F15-EX48F15
BG41F15-BG48F15
Date: July 8th, 2015
Etching start time: 10:40 am
Etching finish time: 11:40 am
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#7</u>

- Time – High – 105 minutes
- Temp. – Low - 50°C
- Molarity – High – 3.0 M
- PH – Low – 7

Films used:
EX49F15-EX56F15
BG49F15-BG56F15
Date: July 8th, 2015
Etching start time: 1:32 pm
Etching finish time: 3:17 pm
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#8</u>

- Time – Low – 60 minutes
- Temp. – High - 65°C
- Molarity – High – 3.0 M
- PH – Low – 7

Films used:
EX57F15-EX64F15
BG57F15-BG64F15
Date: July 9th, 2015
Etching start time: 10:02 am
Etching finish time: 11:02 am
First rinse time: 45 minutes
Second rinse time: overnight

<u>Experiment 1-#9</u>

- Time – High – 105 minutes
- Temp. – High - 65°C
- Molarity – High – 3.0 M
- PH – Low – 7

Films used:
EX65F15-EX72F15
BG65F15-BG72F15
Date: July 9th, 2015
Etching start time: 11:55 am

Etching finish time: 1:40 pm
First rinse time: 45 minutes
Second rinse time: overnight

Experiment 1-#10

- Time – Low – 60 minutes
- Temp. – High - 65°C
- Molarity – Low – 2.0 M
- PH – High – 3

Films used:
EX73F15-EX80F15
BG73F15-BG80F15
Date: July 9th, 2015
Etching start time: 3:00 pm
Etching finish time: 4:00 pm
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 1-#11

- Time – High – 105 minutes
- Temp. – Low - 50°C
- Molarity – Low – 2.0 M
- PH – High – 3

Films used:
EX81F15-EX88F15
BG81F15-BG88F15
Date: July 10th, 2015
Etching start time: 9:37 am
Etching finish time: 11:22 am
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 1-#12

- Time – Low – 60 minutes
- Temp. – High - 65°C
- Molarity – High – 3.0 M
- PH – High – 3

Films used:
EX89F15-EX96F15
BG89F15-BG96F15
Date: July 13th, 2015
Etching start time: 10:04 am
Etching finish time: 11:04 am
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 1-#13

- Time – High – 105 minutes
- Temp. – High - 65°C
- Molarity – Low – 2.0 M
- PH – High – 3

Films used:
EX97F15-EX104F15
BG97F15-BG104F15
Date: July 13th, 2015
Etching start time: 11:40 am
Etching finish time: 1:25 pm
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 1-#14

- Time – High – 105 minutes
- Temp. – Low - 50°C
- Molarity – High – 3.0 M
- PH – High – 3

Films used:
EX105F15-EX112F15
BG105F15-BG112F15
Date: July 14th, 2015
Etching start time: 9:33 am
Etching finish time: 11:18 am
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 1-#15

- Time – Low – 60 minutes
- Temp. – Low - 50°C
- Molarity – High – 3.0 M
- PH – High – 3

Films used:
EX113F15-EX120F15
BG113F15-BG120F15
Date: July 14th, 2015
Etching start time: 1:25 pm
Etching finish time: 2:25 pm
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 1-#16

- Time – High – 105 minutes
- Temp. – High - 65°C
- Molarity – High – 3.0 M

- PH – High – 3

  Films used:
  EX121F15-EX128F15
  BG121F15-BG128F15
  Date: July 13th, 2015
  Etching start time: 1:35 pm
  Etching finish time: 3:20 pm
  First rinse time: 30 minutes
  Second rinse time: overnight

Upon completion of the initial screening experiment, a second factorial experiment was designed. The factor levels chosen for this experiment were as follows:

- A – Time → High – 75 minutes
  Low – 60 minutes
- B – Molarity → High – 2.5 M
  Low – 2.0 M
- C – Temperature → Held constant at 65°C
- D – PH → Held constant at 3
- Films requiring exposure were exposed with Am-241 source K9-744 for 15.8 minutes, using the CAIRS dosimeter head apparatus.

<u>Experiment 2-#1</u>

- Time – Low – 60 minutes
- Temp. – Constant - 65°C
- Molarity – Low – 2.0 M
- PH – Constant – 3

  Films used:
  EX01G15-EX08G15
  BG01G15-BG08G15
  Date: July 23rd, 2015
  Etching start time: 2:12 pm
  Etching finish time: 3:12 pm
  First rinse time: 30 minutes
  Second rinse time: overnight

<u>Experiment 2- #2</u>

- Time – High – 75 minutes
- Temp. – Constant - 65°C
- Molarity – Low – 2.0 M
- PH – Constant – 3

  Films used:
  EX09G15-EX16G15
  BG09G15-BG16G15
  Date: July 24th, 2015
  Etching start time: 9:36 am

Etching finish time: 10:51 am
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 2-#3

- Time – Low – 60 minutes
- Temp. – Constant - 65°C
- Molarity – High – 2.5 M
- PH – Constant – 3

Films used:
EX17G15-EX24G15
BG17G15-BG24G15
Date: July 24th, 2015
Etching start time: 10:59 am
Etching finish time: 11:59 am
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 2-#4

- Time – High – 75 minutes
- Temp. – Constant - 65°C
- Molarity – High – 2.5 M
- PH – Constant – 3

Films used:
EX25G15-EX32G15
BG25G15-BG32G15
Date: July 27th, 2015
Etching start time: 9:34 am
Etching finish time: 10:50 am
First rinse time: 30 minutes
Second rinse time: overnight

With the factor effects narrowing down, a third factorial experiment was designed. For the third factorial experiment, the factor parameters chosen were as follows:

- A – Time → High – 80 minutes
  Low – 70minutes
- B – Temperature → High - 65°C
  Low - 55°C
- C - Molarity → Held constant at 2.5 M
- D – PH → Held constant at 3
- Films requiring exposure were exposed with Am-241 source K9-744 for 15.8 minutes, using the CAIRS dosimeter head apparatus.

Experiment 3-#1

- Time – Low – 70 minutes

- Temp. – Low - 55°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
EX01G215-EX08G215
BG01G215-BG08G215
Date: August 4th, 2015
Etching start time: 1:15 pm
Etching finish time: 2:25 pm
First rinse time: 30 minutes
Second rinse time: overnight

<u>Experiment 3-#2</u>

- Time – High – 80 minutes
- Temp. – Low - 55°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
EX09G215-EX16G215
BG09G215-BG16G215
Date: August 4th, 2015
Etching start time: 2:40 pm
Etching finish time: 4:00 pm
First rinse time: 30 minutes
Second rinse time: overnight

<u>Experiment 3-#3</u>

- Time – Low – 70 minutes
- Temp. – High - 65°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
EX17G215-EX24G215
BG17G215-BG24G215
Date: August 6th, 2015
Etching start time: 9:35 am
Etching finish time: 10:55 am
First rinse time: 30 minutes
Second rinse time: overnight

<u>Experiment 3-#4</u>

- Time – High – 80 minutes
- Temp. – High - 65°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
EX25G215-EX32G215
BG25G215-BG32G215
Date: August 6th, 2015
Etching start time: 12:05 pm
Etching finish time: 1:15 pm
First rinse time: 30 minutes
Second rinse time: overnight

In the fourth experimental design, a regression model was fitted to previous data, guiding the choices of factor settings. By utilizing statistical analysis techniques, a response surface was constructed using prior data, and with the predictions of the regression model, the parameter values for subsequent experiments were determined.

- Films requiring exposure were exposed with Am-241 source K9-744 for 15.8 minutes, using the CAIRS dosimeter head apparatus.

Experiment 4-1+Δ

- Time → 80:12 minutes
- Temperature → 66°C
- Molarity → 2.5 M
- PH → 3

Films used:
EX01H15-EX08H15
BG01H15-BG08H15
Date: August 12th, 2015
Etching start time: 9:45 am
Etching finish time: 11:06 am
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 4-2+Δ

- Time → 80:24 minutes
- Temperature → 67°C
- Molarity → 2.5 M
- PH → 3

Films used:
EX09H15-EX16H15
BG09H15-BG16H15
Date: August 12th, 2015
Etching start time: 11:55 am
Etching finish time: 1:16 pm
First rinse time: 30 minutes
Second rinse time: overnight

Experiment 4-3+Δ

- Time → 80:36 minutes
- Temperature → 68°C
- Molarity → 2.5 M
- PH → 3

Films used:
EX17H15-EX24H15
BG17H15-BG24H15
Date: August 12th, 2015
Etching start time: 2:17 pm
Etching finish time: 3:34 pm
First rinse time: 30 minutes
Second rinse time: overnight

The data collected from the previous three experimental runs suggested that the optimal point on the response surface had already been reached, as the quality of the films had actually deteriorated, relative to previous experimental parameters. For this reason, further experimenting was necessary. It was decided upon that, by considering the previous data sets, observance of lower time parameters, including 50 and 55 minutes, would be ideal for further exploration of the optimal etching procedure. As such, two more experiments were run at the lower times.

- Films requiring exposure were exposed with Am-241 source K9-744 for 15.8 minutes, using the CAIRS dosimeter head apparatus.

<u>Experiment 5-#1</u>

- Time – High – 50 minutes
- Temp. – High - 65°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
EX25H15-EX32H15
BG25H15-BG32H15
Date: August 18th, 2015
Etching start time: 9:45 am
Etching finish time: 10:35 am
First rinse time: 30 minutes
Second rinse time: overnight

<u>Experiment 5-#2</u>

- Time – High – 55 minutes
- Temp. – High - 65°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:

EX33H15-EX40H15
BG33H15-BG40H15
Date: August 6th, 2015
Etching start time: 1:15pm
Etching finish time: 2:10pm
First rinse time: 30 minutes
Second rinse time: overnight

In order to conclude experimentation, the final step was to run a few commissioning test etches to determine the precision of repeated etching under the new, optimized procedure.

Commissioning-#1

- Time - 60 minutes
- Temp. – High - 65°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
EX41H15-EX44H15
BGBLANK-BGBLANK
CHECK#1-#4
Date: August 20th, 2015
Etching start time: 10:16am
Etching finish time: 11:16am
First rinse time: 30 minutes
Second rinse time: overnight

Commissioning-#2

- Time - 60 minutes
- Temp. – High - 65°C
- Molarity – Constant – 2.5 M
- PH – Constant – 3

Films used:
CHECKBLANK
BG
Date: August 26th, 2015
Etching start time: 2:12pm
Etching finish time: 3:12pm
First rinse time: 30 minutes
Second rinse time: overnight

9. Analysis

To commence the experiment, the film exposing procedure was worked out. First the time needed to theoretically produce 1005 tracks, using sources K9-744 (Am-241, 1025Bq), H6-989 (Am-241, 670Bq), and FP-227 (Am-241, 3030Bq), was calculated (see Appendix). In addition to the three choices of sources, different exposure apparatus were used, each with different geometric efficiencies. The following table contains the data used to inform the exposing procedure decisions.

Table 1: Exposure procedure test

Test #	Source (Bq)	Time (minutes)	Apparatus	Registration Efficiency
1	3030	9.5	Etch check	38.6%
2	3030	9.5	Etch check	37.9%
3	670	24.3	Dosimeter head	73.5%
4	670	24.3	Dosimeter head	54.2%
5	1025	27.0	Etch check	30.6%
6	1025	15.8	Dosimeter head	61.1%
7	3030	5.5	Dosimeter head	62.7%
8	3030	5.5	Dosimeter head	49.4%
9	1025	15.8	Dosimeter head	62.3%
10	1025	15.8	Dosimeter head	57.3%
11	670	24.3	Dosimeter head	52.1%
12	670	24.3	Dosimeter head	27.6%

The table shows an important quality of the choice of exposing apparatus. The dosimeter head has a much better geometric efficiency, $\varepsilon_G = 0.001037$, compared to $\varepsilon_G = 0.000601$ for the etch-checking apparatus. This has the effect of decreasing the amount of time needed for exposures, in the case of the dosimeter head, and increasing the exposure time required in the case of the etch-checking apparatus. Among other considerations, this was a potent motivator for choosing the dosimeter head as the exposure apparatus for the experiments. Upon analysis of data received in dosimeter head related tests, it was observed that when taking an average of the registration efficiency achievable with each distinct source, the 1025 Bq source was most consistent, as well as highest achieving, at an average of 60.2% efficiency. The final reason for choosing the CAIRS dosimeter head as the exposing apparatus is that the RSIC actually uses this apparatus in their professional dosimetry service. For these reasons, the exposing regime was finalized, at source K9-744 1025 Bq for 15.8 minutes, using the CAIRS dosimeter head.

With the exposing regime decided upon, the next step was to calculate the necessary sample size to achieve statistically significant results (see Appendix). Using prior RSIC etch check film data, the standard deviation for the sample size calculation was estimated to be $\sigma=0.071562$. A 95% confidence level was desired for the experiment, which corresponds to an error value of E=0.05, as well as a Z-score value (from a z-table) of $z_{\alpha/2}$= 1.96. Using these values in the calculation of sample size, it was determined that a sample size of 8 pieces of film would yield significant data. To this end, 8 exposed pieces of film and 8 background films were necessary for each batch of experimenting. With the sample size calculated, and the exposing procedure determined, the experiment was commenced.

The factors explored in this experiment were etching time, etching temperature, molarity of etching solution, and PH of rinsing baths. The quality of the film etching, no doubt, varies with numerous factors beyond these. The initial experimental design was a two-level, four-factor factorial designed experiment. A factorial approach to the experimental design was taken as it was deemed most efficient for screening for effectiveness of the chosen factors. The factorial design is realized by choosing a high and low level for each parameter, and then varying each factor in combinations of high and low levels of the other factors, until all of the possible combinations have been used. In the following charts, a "+" sign indicates that a factor was at its high level, while a "-"indicates the factor was at its low level.

The motivations for the specific parameter values of each factor in the initial screening experiments were due to several reasons. In an article by Nikezić and Janićijević (2002) it was noted that the removed layer of LR115 detectors increased linearly with etching time. A figure given in the aforementioned paper, with isothermal lines of various temperatures, was used to inform the decisions regarding time and temperature parameters. Also, the figure qualitatively showed the degradation of the film's emulsion layer to an unusable state for etching times longer than 110 minutes, under certain temperature constraints. Furthermore, etches longer than 110 minutes were deemed undesirable from an economic standpoint, as the increased time to etch would place added stress on an already time demanding process. The choice of a lower time parameter value of 60 minutes was based simply on intrigue, as it was completely unknown whether a 60-minute etching procedure would be capable of producing results. Similarly, the parameter levels of 3.0 M and 2.0 M etching solution were chosen as a means of testing for an upper and lower bound on molarity of etchant, relative to the current etchant molarity of 2.5 M. The choice to test the effects of an acidified rinse bath, of PH 3, was due to a personal correspondence via email between former RSIC manager Brian Bjorndal and the then manager of DOSIRAD France, Jean Andru. In the correspondence, Mr. Andru describes the rinsing of LR115 in an acidified bath at around PH 3, to quickly neutralize the attack of the NaOH solution. The choice of rinsing time of 30 minutes was made with consideration of comments made in the previously mentioned email (Andru, written communication). Finally, the parameter levels of 50°C and 65°C for the temperature factor were chosen as lower and upper bounds on the initial experiment, respectively, on consideration of the article by Nikezić and Janićijević (2002). The data obtained from Experiment #1 is shown on the attached spreadsheet (see Table 2).

Sample calculation of registration efficiency, ε_R:

$$\varepsilon_R = \frac{\#\alpha\ measured}{\#\alpha\ expected} = \frac{N_{counts} * \kappa_{IAS}}{A * t * \varepsilon_G} * 100$$

$Ncounts = 189 \pm 5\%$, $\kappa_{IAS} = 4.68535$, $A = 1022.3Bq \pm 30.7Bq$, t=948$s \pm 5s$, $\varepsilon_G = 0.001037$

$$\varepsilon_R = \frac{189 * 4.68535}{1022.3Bq * 948s * 0.001037} * 100 = 88.1126754\%$$

$$\delta\varepsilon_R = \delta\left(\frac{N_{counts} * \kappa_{IAS}}{A * t * \varepsilon_G}\right)$$

$$\delta\varepsilon_R = \sqrt{\left(\frac{\delta N_{counts} * \kappa_{IAS}}{A * t * \varepsilon_G}\right)^2 + \left((N_{counts} * \kappa_{IAS})\left(\left(\frac{\delta A * t * \varepsilon_G}{(A * t * \varepsilon_G)^2}\right) + \left(\frac{A * \delta t * \varepsilon_G}{(A * t * \varepsilon_G)^2}\right)\right)\right)^2} * 100$$

$\delta\varepsilon_R$ =5.392842302%

$\varepsilon_R = 88.1\% \pm 5.4\%$

The data from the initial experiment shows a wide variety of film etching conditions, and results. By using a factorial design, as can be seen, many combinations of parameters of factors were explored. However, only some of the etching batches produced usable results. In batches 1, 5, 6, 7, 11, 14, and 15, the analysis showed that the films had been under-etched. As a result, the films either yielded no counts at all, or far fewer counts than the number that was expected. In batches 8, 9, 12, and 16, the analysis showed that the films had been over-etched. This resulted in unusable films that were extremely difficult to read, or else produced erroneous results due to the internal settings of the image acquisition system (IAS).

Batches 2, 3, 4, 10, and 13 were deemed usable, upon analysis with the IAS. The analysis shows that the registration efficiency varies most with etching temperature, considerably with etching time, less with molarity, and least with the PH of the rinse. The lowest registration efficiency achieved was 48.01% in batch 2, while the highest was 80.19% in batch 10. Batch 2 involved etching for 105 minutes, at 50°C, with a molarity of 2.0 M, and a rinse in tap water. Batch 10 concerned etching for 60 minutes, at 65°C, with a molarity of 2.0 M, and a rinse in acidified water at PH 3. As well, the average background count for batches 2 and 10 were 0 and 3, respectively. Among the etching batches in this experiment, batch 10 achieved the highest registration efficiency, with minimal background, and the best signal-to-background ratio of all the batches. Since data was limited by under and over-etching, this piece of data was interpreted as important, as it demonstrated the effects of temperature on the quality of the film etches. An analysis of variance (ANOVA) chart for this experiment is attached. Because of the success of the batch 10 parameters, the follow up experiment was designed to explore the parameter regions near the batch 10 settings.

In the second experiment, the choices of parameter levels were based on the results of the screening experiment. The goal of the second experiment was to show the effects of molarity and etching time on film quality, as the temperature and PH settings had previously been shown to produce quality results. It was noted that although there are several holes in the data of the initial experiment, the highest registration efficiency achieved was 80.19% efficiency, with an average background count of 3, and a signal/background ratio of 267.3. This result was attained in batch 10, with a temperature of 65°C, a PH of 3, and the time and molarity parameters on lows of 60 minutes and 2.0 M, respectively. The data obtained is shown on the attached spreadsheet (see Table 3). Because the screening experiment had shown both desirable and undesirable factor combinations, the scope of the second experiment was made narrower by decreasing the size of the factorial design, focussing more on the exploration of relevant factors to film etching quality. For this experiment, the PH of the rinse bath was held constant at 3, the temperature of the etching bath held at 65°C, while the time was allowed to vary between 60 and 75 minutes. As well, the molarity of the etchant was varied between 2.0 M and 2.5 M. The registration efficiency value of 80.19% calculated in batch 1 indicates the reproducibility of results obtained in batch 10 of the initial experiment. The efficiencies obtained from the remaining batches, including 82.05% in batch 2, 79.72% in batch 3, and 73.66% in batch 4, were comparable to batch 1. This result seemed to confirm the predictions that temperature was the most important factor to film quality, but the most intriguing result was in the background data. The background counts on average for each batch were 3, 4, 2, and 5 in batches 1, 2, 3, and 4, respectively. A linear trend in background counts, despite the random nature of background radiation, appears to be correlated with longer etching times, and less so with increasing molarity. The results seem to point to lower etching times to decrease the overall number of background tracks, while the average registration efficiency achievable does not seem to vary much with etching time or molarity, at a constant temperature. A closer look at the data from this experiment can be found in the attached ANOVA spreadsheet. It was decided that another experiment could be done to more thoroughly explore the interplay between etching time and etching temperature, and a third experiment was designed.

The levels for each factor in the third experiment were chosen based on results from the first and second factorial experiments. Since it was apparent that molarity and PH had minor effects on film quality, those factors were kept constant. Temperature and time had the most drastic effect on the film quality, and were therefore investigated more closely with this experiment. The experiment yielded the data shown on the attached spreadsheet (see Table 4). In batch 1, an efficiency of 61.54% was obtained, with an average background count of 1. This results in an excellent signal/background ratio. However, the registration efficiency is too low for the required accuracy of the RSIC's dosimetry service. A similar result was obtained in batch 2, where the time parameter was increased by 10 minutes, from 70 minutes to 80 minutes. The first two batches, differing by only 10 minutes of etching time, seem to produce essentially the same results. In batch 3, an increase in temperature resulted in an increase in registration efficiency, up to 72.62%. However, the background counts also saw an increase, up to an average of 5. Finally, in batch 4, the background count increased further, to 8 counts, and the efficiency also improved to 88.55%. In this case, the films were very close to being over-etched, and the increased efficiency is mostly attributable to the threshold of the counting system. For this reason, the factor settings of batch 4 were determined to be undesirable. The batches from this experiment show a clear interaction between the time and temperature factors. While increasing time and temperature allows a larger amount of tracks to be counted by the IAS, the number of background counts also increases, reducing the overall signal/background ratio. An ANOVA spreadsheet is attached, which displays the data in a more analysis-effective way. The ANOVA data was used to construct response surfaces, displaying the statistics behind the effects of the factor settings. The response surfaces shown in the ANOVA spreadsheets show that, in general, an increase in time and temperature should increase counts, along with background counts. A regression model to explain the behaviour of the counts as a function of etching time and temperature was developed, and it was determined to be $143.103125+3.309375x_1+15.496875x_2$. Accordingly, the background data was fitted to a regression model in a similar way, and it was calculated to be $3.59375+0.84375x_1+2.90625x_2$. The variables are coded, and do not carry physical units. The variables are representative of the settings of the time and temperature parameters. The regression models were used to determine the settings that the iterations of subsequent experimenting should take on. Upon completion of this third factorial experiment, the necessary data for an optimization experiment had been taken. By analyzing and paying special attention to the ANOVA data from this experiment, a fourth experiment was designed to investigate the properties of film etching along the curve given by the regression model.

The levels used in the fourth experiment were chosen to follow the gradient of the response surface. It is believed that in an ideal situation, the gradient of the response surface can be followed in such a way as to improve the desired response. The desired response was to increase efficiency while decreasing background counts. The difficulty in achieving the desired response is that the parameters that contribute to increased efficiency also increase the background counts. Thus, a balancing of factors had presented itself. The batches in experiment 4 were intended to observe this balancing of parameters, and the data obtained is shown on the attached spreadsheet (see Table 5). The origin point of the experiment was chosen to coincide with the previously desirable factor levels, as well as the gradient of the response surface. Then, the factor levels were increased in specific increments, along the gradient of the response surface. The hope was to realize when the optimal point had been reached by watching for increases or decreases in the response. From the data taken, it appeared as though the optimal point had already been reached. A look at batch 1 shows the efficiency was 78.25% with a background of 6, which had increased the background counts from previous experiments, while leaving the efficiency relatively unchanged. For this reason, the experiment was concluded short, as no further experiments in this manner would yield the optimal result. However, it was unclear whether a shorter time parameter would potentially give a better signal/background ratio. Therefore, a fifth experiment was designed to investigate the lower time parameters.

The fifth experiment produced the data shown on the attached spreadsheet (see Table 6). In this experiment, 2 batches were made, each with different time variations. The data from these batches shows the importance of the time parameter values, as the lower times had decreased the background count considerably. The downside to the time parameters chosen was the decrease in efficiency noticed in comparison to previous time settings. Therefore, it was deduced that the optimal etching procedure had already been attained in previous experiments. The parameters chosen to represent the optimal etching procedure at this point were 60 minutes for time, at 65°C, with a molarity of 2.5 M, and a rinse in a water bath of PH 3. The final step in experimenting was to run a few commissioning tests, to determine the validity of the conclusions drawn from the previous experiments.

To begin the commissioning batches, exposures of films using both the 670 Bq source, using the etch check apparatus, and the 1025 Bq source, with the CAIRS dosimeter head, were required. The data from these batches is shown on the attached spreadsheet (see Table 7). In the first batch, 4 exposed films from the dosimeter head procedure, and 4 films from the etch check procedure were included, to give an idea of the consistency of the results. As well, 8 background films were included in this batch. The average background count obtained from this first batch was 3, which was expected from previous experiments at this setting. The efficiency obtained for the dosimeter head exposures was 76%, which is, although slightly lower, fairly similar to what would be expected based on prior experimenting. The data from the etch check apparatus exposures shows an efficiency value of 93%. This efficiency is a very good result for the purposes of the RSIC. In batch 2, an average background of 2 was recorded, with an efficiency value of 93%. This is in agreement with the first batch, which increases the validity of the previous measurements.

The data taken in the commissioning tests were used to test the hypothesis that the mean counts, and therefore registration efficiency, of the previous etching procedure are equal to those of the new etching procedure. To test this with the proper statistical significance, the necessary equations were taken from a text by Montgomery (2009). To make a two-way hypothesis test of the difference in means, the following calculations were made:

1. The sample variance of both the etch check log data from the past 12 months, and the commissioning data, was calculated (see Appendix).

2. The sample variances were used to calculate the pooled estimator of s^2, s_p^2 (see Appendix).
3. Using the pooled estimator, the hypothesis was tested with a t-Test.

Sample Calculation of t-Test statistic, t_0:

$$t_0 = \frac{\overline{x_1} - \overline{x_2} - \Delta_0}{s_p\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}$$

$$t_0 = \frac{813.4639 - 706.7070}{116.8750585 * (0.306186218)}$$

$$t_0 = 2.983241932$$

$$t_0 = 2.983$$

Using this value, the hypothesis that the two means from previous data and the commissioning data are statistically equal can be tested. To do so, a t-table was consulted and the following comparisons were made:

$H_0: \mu_1 - \mu_2 = \Delta_0$, the hypothesis that the means are equal, corresponding to $\Delta_0 = 0$.

$H_1: \mu_1 \neq \mu_2$, the alternative hypothesis

$t_{0.025,106} = \pm 1.9846667$

Reject the null hypothesis if $-t_{0.025,106} < t_0 < t_{0.025,106}$

-1.9846667<2.983≮ 1.9846667

As a result of this comparison, the null hypothesis, that the mean counts of both etching procedures are equal, must be rejected. This result will be discussed thoroughly in the concluding section of this article.

Next, the same procedure must be undertaken to compare the background data from the RSIC's previous data and the commission testing results, except a one-sided hypothesis test will be employed:

$H_0: \mu_1 = \mu_2$, the hypothesis that the means are equal

$H_1: \mu_1 > \mu_2$, the alternative hypothesis

$t_{0.5,74} = \pm 1.66796667$

$t_0 = -0.523407774$

Reject the null hypothesis if $t_0 > -t_{0.5,74}$

The test succeeds in rejecting the hypothesis that the mean background of the old procedure was equal to that of the new procedure. Therefore, the alternative hypothesis that the new procedure reduces the background counts is accepted. Another test of significance is to compare the variance of the two procedures.

$H_0: \sigma_1^2 = \sigma_2^2$

$H_1: \sigma_1^2 > \sigma_2^2$

$F_{0.05,n_{59},n_{15}}$ =2.162

$$F_0 = \frac{s_1^2}{s_2^2} = \frac{55.8813559}{7.6666667} = 7.288872477$$

Reject this hypothesis if $F_0 > F_{\alpha,n_{1-1},n_{2-2}}$

The null hypothesis must be rejected as a result of this test. The alternative conclusion is that the variance in the RSIC background data is higher than the commission testing background data. This is a successful result, as a minimized and more precise background count will improve the quality of the dosimetry service.

10. Conclusions and Recommendations

In Experiment #1, a wide variety of etching conditions were employed. The purpose of the experiment was to give an initial idea of the effects each of the four factors – time, temperature, molarity and PH – may have on the quality of film etching. This question is of utmost importance to the RSIC; the quality of the films, as well as the accuracy of the data obtained from them, relies heavily on the etching conditions. The initial experiment was successful, in the sense that it displayed many undesirable etching conditions, which could quickly be disregarded, along with a few desirable etching parameters which could be explored more thoroughly in subsequent experiments. One such set of etching conditions was obtained in batch 10, where the registration efficiency calculated was 80.19% with an average background count of 3. This particular batch was the motivation for the parameters in the second experiment. In experiment #2, the focus was on the effects of time and molarity, as it was apparent after the first experiment that the PH of the rinse was not majorly important, although most likely a good idea, while the temperature was confirmed to be a large contributor to film quality. With the temperature held at 65°C, and a rinse bath held at 3 PH, the data obtained from experimenting showed relatively unwavering results. The efficiency calculated in batch 1 was 80.65%, with a background count of 3, which was expected from the previous experiment, under the same etching conditions. The second batch here improved the efficiency, up to 82.05%, with an increase in the background counts, up to 4, as well. The remaining batches here showed decreased registration efficiency, as the films became more etched away, and an increase in the background counts as well. The success of this experiment was to show that etching time has a noticeable effect on the background counts, and a saturation effect in the registration efficiency. The third factorial experiment was conducted to more closely observe the interplay between etching time and temperature. It was made apparent by the data collected that the time parameter had a drastic effect on the background counts obtained, with increasing temperatures. The temperature parameter had the most effect on the registration efficiency achieved. In combination, a longer etching time at a higher temperature had the highest registration efficiency, at 88.55%, with a high background count of 8. As a result of the previously attained data, a response surface was constructed using ANOVA data. The course of action taken was to utilize the regression model predicted by the ANOVA data to follow the gradient of the response surface in the direction of increasing efficiency, while attempting to minimize the background counts. The data shown in Table 5 shows the experiments conducted to this end. From this experiment, it was predicted that the optimal etching procedure had already been realized in previous experiments, as the data presented a slight decrease in registration efficiency, with a notable increase in background counts. For the purposes of argument, two more batches were made in a fifth experiment, using shorter etching times. This resulted in a decrease in registration efficiency with a drastic decrease in background counts. An advantage to conducting the experiments this way was that having data on both ends of the spectrum, longer etching times and shorter etching times, gave validity to the prediction that an etching procedure at 65°C, for 60 minutes, with molarity of 2.5 M, and a rinse in PH 3 water, would produce the best results. As such, commissioning tests were conducted. In the commissioning tests, two batches were made at the agreed upon parameters. The films used for exposure by the 670 Bq source and etch check apparatus used by the RSIC gave an average registration efficiency of 93%. As well, the average background counts obtained across both batches was 2. These results are quite good, and the precision of the results of this etching procedure is desirable. Once the commissioning tests had been concluded, the statistical comparisons of the old etching procedure to the new one were made. The necessary testing statistic was calculated to be 2.983. In comparing the registration efficiency of the old procedure to the new one, it was found that the hypothesis test for the equality of the means failed. This is reasonable for a few reasons. The variability in the etch check log data is erratic. From the last 12 months at the RSIC, the average counts obtained from etch check films were higher than the expected counts. Technically, this should not be possible, although radiation processes are inherently random. For this reason, the RSIC has achieved actually better than 100% registration efficiency in some cases. For this to indeed be the case, there must be other factors contributing to this result, such as IAS settings, thickness of film received from DOSIRAD, etching variability, etcetera. With the procedure proposed in this paper, the variability of measurements has decreased, even if the registration efficiency was sacrificed somewhat. As there are pros and cons for either process, one advantage to the new process is the precision of the results. The new etching procedure gives more consistent registration efficiency results. As well, the results given by the new etching procedure can be interpreted as more “physically” realistic. A similar comparison was made between the commission testing background data and the RSIC background log. The statistic used to test the equality of the means was calculated to be -0.523407774. Because this statistic met the rejection criteria, the null hypothesis and the alternative hypothesis was accepted. This shows that the new procedure reduces the background counts. As well, the test for the equality of the variances of the two procedures failed. The alternative conclusion in this case is that the variance of the new procedure is reduced in comparison to the old procedure. The results of the statistical analysis show that the new etching procedure is less variable, with a lower background, and a more realistic display of the radiation exposure of the films used in the RSIC's dosimetry service. For these reasons, the new etching procedure could be adopted by the RSIC to improve the reliability of the services provided. Although the experiment was successful in providing insight into the effects of etching conditions on film quality, much more exploration could be done to improve the dosimetry service. A recommendation for further studies is the effects of stirring during film etching. The reason for this is that some films showed erratic etching patterns. While most of the films of a batch produced useful results, some were etched away in an unpredictable manner, rendering them unusable. It is hypothesised that the cause of this unusual etching behaviour is due to an inconsistency in etching across the entire film. Therefore, a solution may be to stir the films while etching. Another recommendation is to investigate the effects that film thickness may have on film quality, and how the etching conditions may be varied to account for this. It was noted during the experiment that the film batches used might not always be the quoted 12 μm. In fact, the supplier of the films revealed that the newer film batches are actually closer to 10 μm. The film etching procedure is affected by numerous factors beyond these. This is problematic for delivering accurate results. However, with the experiment conducted here and the knowledge of etching conditions gained, the RSIC is closer to achieving the most optimal etching procedure, and along with it, the most effective dosimetry service it can provide.

11. Sources of Error

Some of the sources of error encountered in the experiment were as follows:

- The variability of the temperature bath. Although the variation in temperature was mostly accounted for with allowed experimental error, there is the chance of inaccuracies in the temperature detection of the thermistor in the heating bath. This could be remedied with a less variable, unbiased thermometer to measure the variations.
- Small variations in the molarity and PH of etching solutions and rinse baths, respectively, also present small amounts of variability. This was mostly accounted for with allowed experimental error.
- The variability of the image acquisition system. It was observed that between films, the image acquisition system took different counts than what were expected, as well as not being standardized in regards to lighting levels, focus, etcetera. As well, the threshold value of the software occasionally

needed to be adjusted to give more realistic counts. The variability here is based on human subjectivity, so removing the chance for a subjective intervention is crucial. The error caused by this was mostly accounted for with allowed experimental error. The process may be remedied by inquiring into the standardization of the image acquisition system.

- The exposures made with the CAIRS dosimeter head were occasionally affected by the fact that the dosimeter head did not seal the films to the collimators every time. As a result, some alpha particles would lose more energy, and thus be detected less accurately, due to extra separation between collimator and film. Remedy this with a tighter fitting exposure device.
- If the films in the experiment varied between 12 micrometers and 10 micrometers, the effects on film quality could be large. The only way to remedy this is to know ahead of time what thickness of film is being used.
- The geometric efficiencies of the exposure apparatus used are quoted differently in some technical documents. Depending on the choice of geometric efficiency, the data could be shifted from what was presented in this experiment. A solution to this problem is to standardize the geometric efficiencies of the etch-checking apparatus.
- The radiation sources used may not be perfectly uniform in shape and size, and therefore the decay counts may not be consistent.