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Subject: Optimization of Etching and Reading Procedures for the Autoscan 60 Track Etch System

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Optimization of Etching and Reading Procedures for the Autoscan 60 Track Etch System

Introduction

The Los Alamos National Laboratory is charged with measuring the occupational exposure to radiological workers and contractors throughout the Laboratory, which includes many different sites with multiple and varied radiation fields. Of concern here are the high energy neutrons such as those generated during accelerator operations at Los Alamos Neutron Science Center (LANSCE).

In the past, workers were monitored using a four element Harshaw TLD in combination with a Kodak NTA type A neutron sensitive film badge. The doses from the two dosimeters were summed, due to the energy dependent response characteristics of the dosimeters, and assigned to the individual wearing them.

While accurate, the NTA dosimeters were subject to track fading, hence had to be assembled in a nitrogen filled glove box, and exchanged monthly. The counting of the films using a Leitz microscope was time consuming, while the above mentioned assembly procedure was quite labor intensive.

In 1993, the Los Alamos National Laboratory purchased an Autoscan 60 automated reader for use with chemically etched CR39 detectors.

The dosimeter design employed at LANL uses a plastic, hemisphereical case, encompassing a polystyrene pyramidal detector holder. The pyramidal holder supports three detectors at a 35° angle. Averaging the results of the three detectors minimizes the angular dependance normally associated with a planar dosimeter.

The Autoscan 60 is an automated reading system for use with CR39 chemical etch detectors. The detectors are immersed in an etch solution to enhance the visibility of the damage sites caused by recoil proton impact with the hydrogen atoms in the detector.

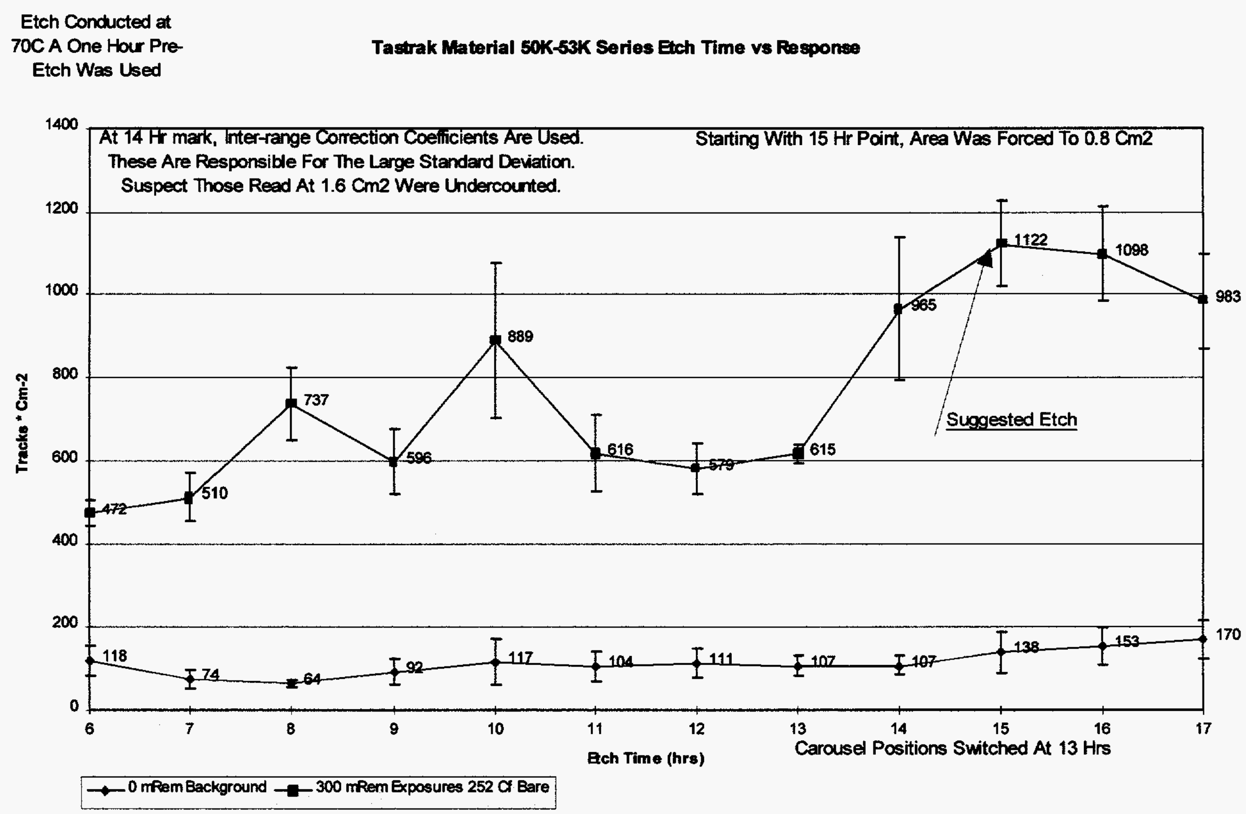
The manufacturer of the Autoscan 60 (NE Technologies), recommends a one hour pre etch in a solution of 60% Methanol and 40% NaOH at 6.25N at 70° C, followed by a six hour etch in NaOH at 6.25N at 70° C . Following etching, detectors are placed in a stop etch solution of HCl at 0.1N for a period of 15 minutes at room temperature (20 °C), followed by a rinse bath of distilled water with a whetting agent at room temperature for an additional 15 minutes. All of the preceding steps are carried out in separate baths equipped with heaters capable of controlling temperatures to within ± 1 °C, when needed, and using agitation. For final cleaning prior to reading the detectors are placed in an ultrasonic bath containing ethanol at room temperature for a period of 10 minutes. Detectors may be dried, then wiped with a soft, lint free cloth.

We decided to increase the etch time from six hours to 15 hours, while retaining the 70° C temperature. The reason for the change in the etch is to enhance the sensitivity and precision of the CR39 detector as indicated by the following study.

Thirty detectors exposed to 300 mRem of ^{252}Cf bare, and thirty background detectors were etched for 1 hour in a 60% methanol / 40 % 6.25N NaOH solution at 70 °C with agitation, then etched for 6 hours in a 6.25N NaOH solution, again at 70 °C with agitation. The detectors were then immersed in a 0.1 N HCl solution as a stop etch, rinsed in water containing a whetting agent, wiped down and read in the Autoscan 60 reader. The process was repeated, with the exception of the pre-etch, on an hourly basis and the results recorded on Chart 1.

As can be seen in Chart 1 below, response in terms of tracks per square centimeter are a function of etch time. The goal here was to maximize response while maintaining a stable and minimal background. A time of 15 hours was selected for a number of reasons. At 15 hours the response is quite high while the curve is relatively flat, thereby reducing the criticality of etch time. Also at 15 hours we may place the foils in the etch bath at the end of the work day, let them etch over night and remove them at the beginning of the following work day for processing.

Chart 1



The next chart (Chart 2) gives the results of a continuous 15 hour etch on foils exposed to 300 mRem of bare 252 Cf, and other foils exposed to background radiation only.

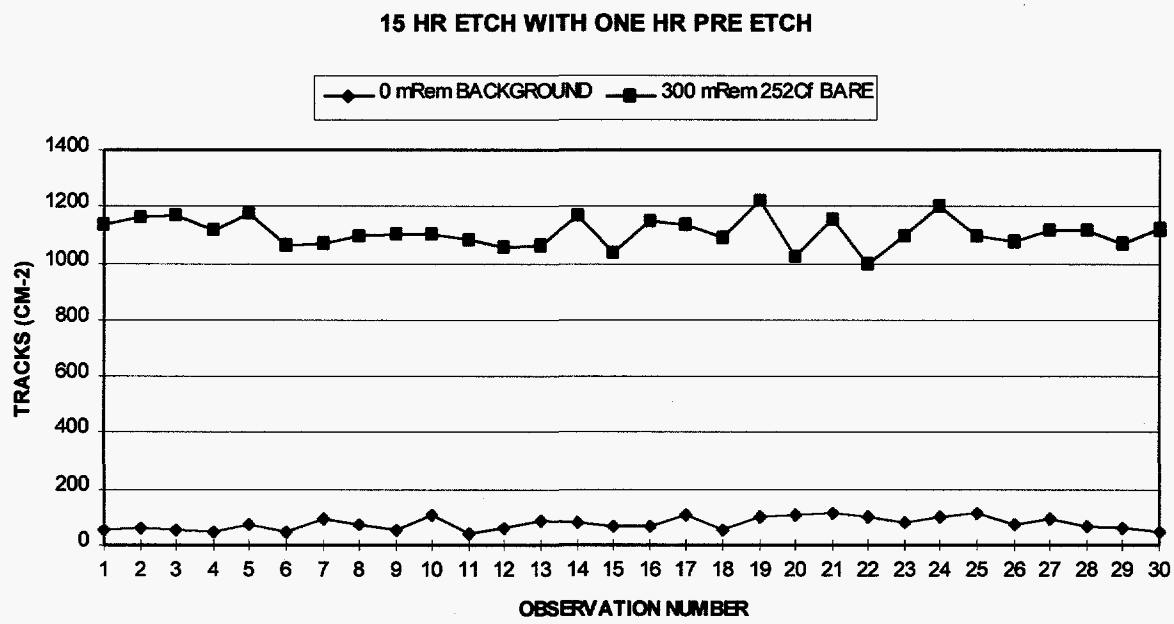


Chart 2

A pre etch, using a solution consisting of 60% Methanol and 40% 6.25N NaOH and lasting one hour is typically carried out at 70°C. The purpose of this pre etch is to rid the detectors of damage sites due to alpha particles and surface damage due to scratching etc.

We decided to attempt an etch process that did not include a pre etch. This was done in order to simplify the process as well minimize the waste stream by eliminating the Methanol/NaOH solution.

Detectors were removed, stop etched, and cleaned hourly, prior to reading.

The results of this run are contained in Chart 3a on the following page.

As shown, the background readings increase to an unacceptably high level while there is the lack of a plateau, or stable point in the 300 mRem data.

Most of the elevated background can be attributed to surface damage, scratching etc., due to excess handling and cleaning of the foils. Testing on uninterrupted etch runs has proved fairly encouraging, yielding a mean background of 95 ± 12 tr*cm-2 and 916 ± 74 tr*cm-2 for a 200 mRem exposure (see Chart 3b). Again these values are for the sum of zone 2 and zone 3 only.

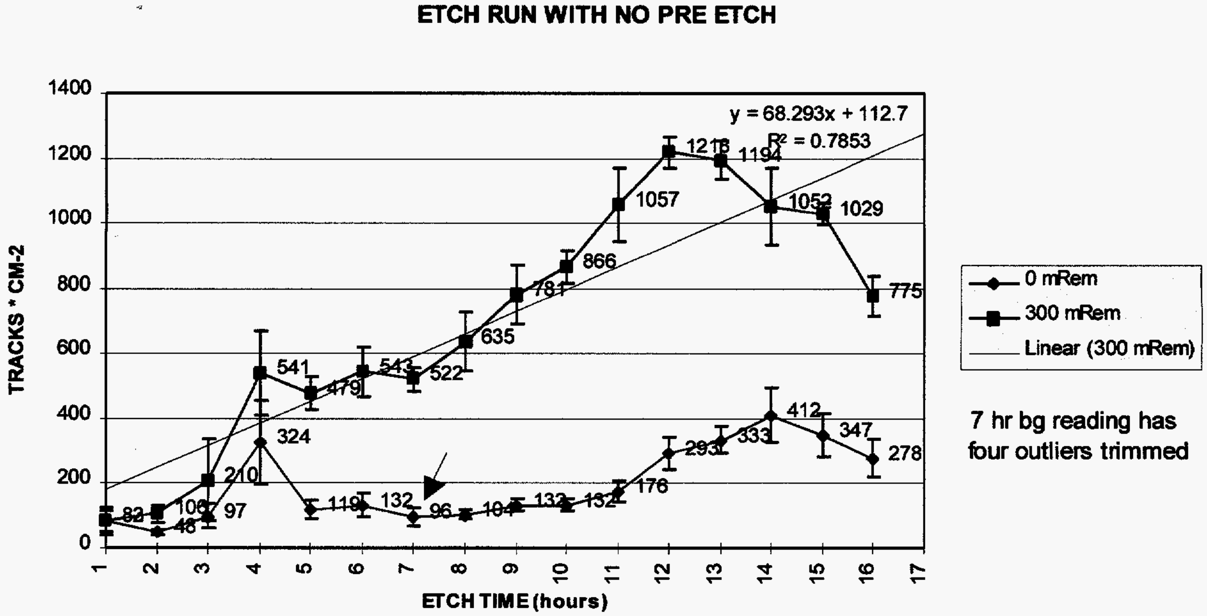


Chart 3a

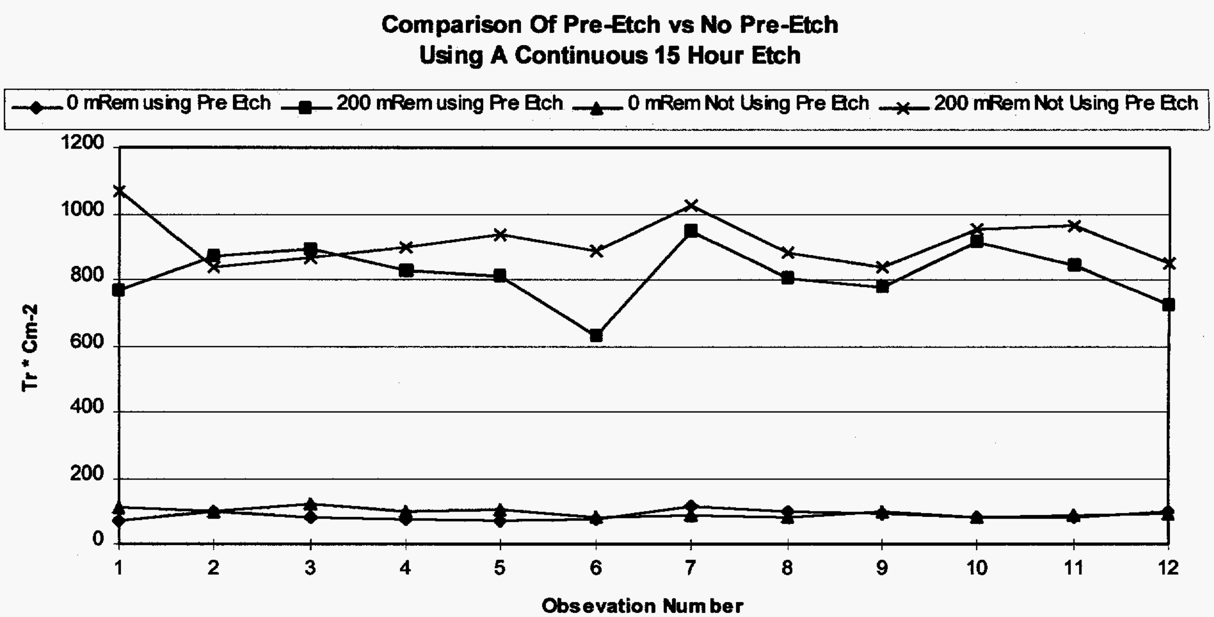
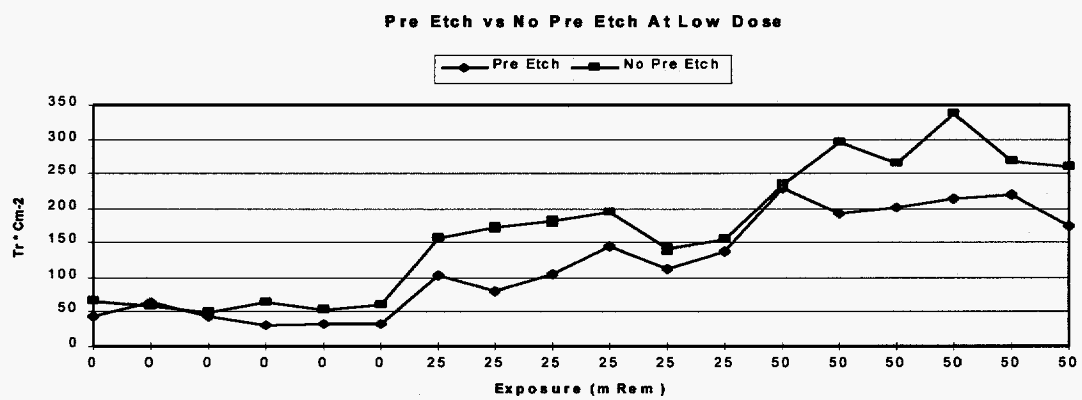


Chart 3b

Further testing has been carried out on lower dose levels with the CR39 material. The results of these tests (see Chart 3c) are consistent with the above preliminary results, therefore we are considering changing our procedure to eliminate the pre etch. This will simplify our waste disposal as well as eliminate a step in the etch process.

Chart 3c



With Pre Etch	mRem	
0	25	50
41 + - 13	114 + - 24	205 + - 20
Without Pre Etch	mRem	
0	25	50
58 + - 6	167 + - 20	277 + - 36

It seems that track or pit diameter is dependant upon etch time and neutron eneregy. With the original six hour etch, using the one hour pre etch our track diameters averaged approximately 10 microns. With the 15 hour etch, with no pre etch, our track diameters average approximately 20 microns. Extending the etch period to 46 hrs, again with no pre etch, gives track diameters of approximately 60 microns, emulating electro chemical etch tracks in size. The above diameters were obtained using bare ^{252}Cf.

We do not yet know what effect these varied etch times may have on energy dependance. We intend to have some detectors exposed to a monoenergetic neutron source beginning at energies in the range of 100 KeV.

In the interim, to check energy dependance, detectors were set up both on phantoms and free in air, and were exposed to 200 mRem of ^{252}Cf bare, D_2O Moderated ^{252}Cf, as well as ^{252}Cf moderated with CH_2 spheres of diameters of 2", 4" and 8". The number of dosimeters used was three on the phantoms and two free in air for each exposure. Once again, each dosimeter holds three detectors.

It may be seen that the response, in terms of gross tracks per cm^{-2}, is quite flat. (See Chart 3d)

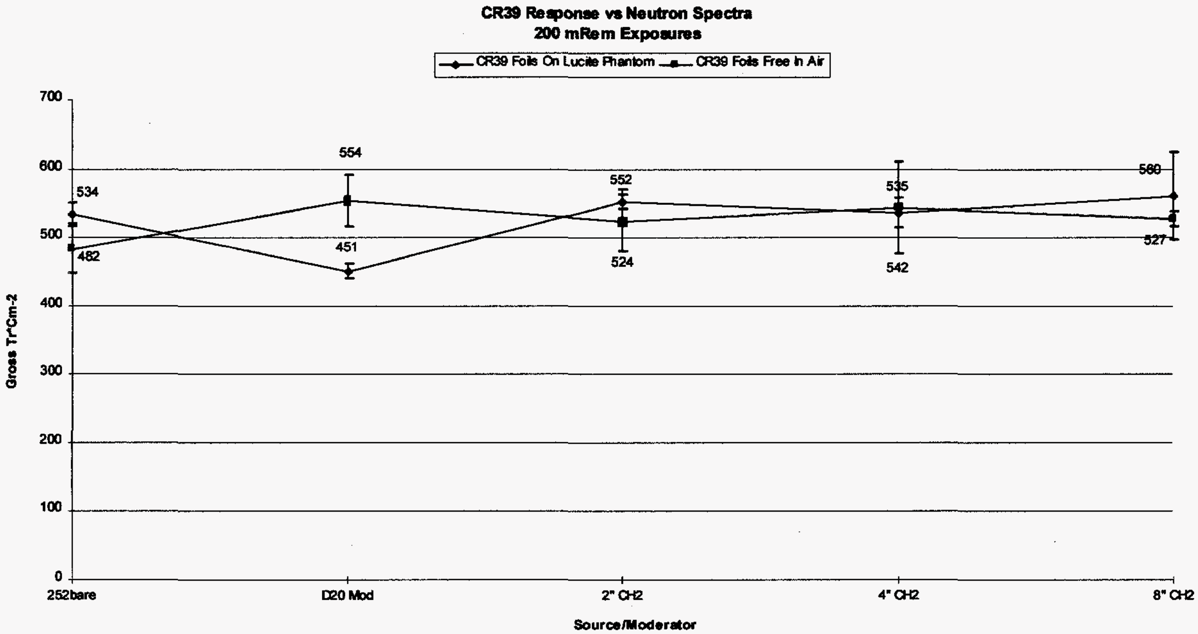


Chart 3d

We feel that the longer etch time, 15 hours, may be responsible for enlarging the track diameter to the extent that they may be counted despite the lower energies associated with the moderated ^{252}Cf. The above data was obtained using the one hour pre etch.

Below (chart 3e), we have plotted track size after a one hour pre etch and 15 hour etch. The track size observations were taken visually using a Lietz Wetzlar microscope at a magnification of 500x. As suspected, track size seems, preliminarily, to be related to neutron energy. We suspect that we may read tracks, reliably, down to approximately ten microns. Further experimentation is required to determine what energy will correspond to an average diameter of ten microns, given a 15 hour etch time. For lower energies, it may be possible to increase etch time in order to enlarge track diameter to a point where the tracks are readable.

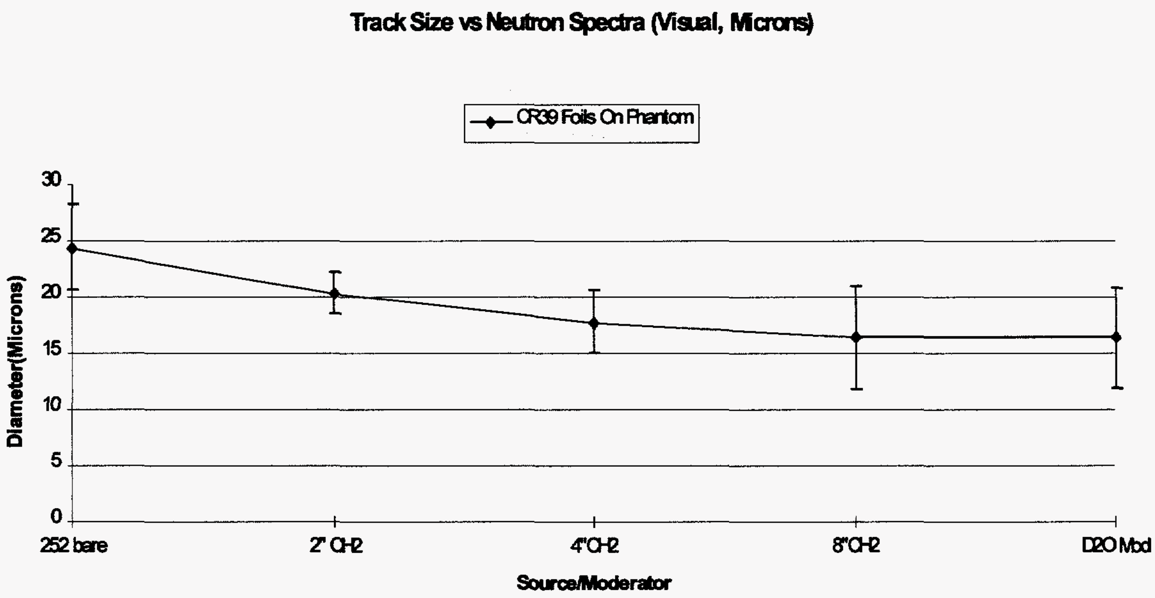


Chart 3e

Also changed was the Zone Bounds in the Parameters data base. The zone bounds are bins into which individual tracks are added based on their size as measured in pixels. As the etch progresses the damage sites become larger and tend to be grouped in the bin reserved for the next largest tracks. The problem is compounded when the system switches magnification due to high track density. Based on this, we increased bounds geometrically with respect to magnification. These new bounds correspond fairly closely to what is recommended by NE Technologies.

Table 1. New Zone Bounds (Pixels)	Area Read	Zone 1 - 2	Zone 2 - 3	Zone 3 - 4
	1.6	2	11	76
	0.8	2	15	100
	0.4	2	22	141
	0.2	2	30	200
	0.1	2	45	280
	0.05	2	60	400

After changing the zone bounds, it became necessary to calculate new inter range correction coefficients and foldback value. Foldback occurs when area switch limits have been exhausted and the system relies on light intensity as measured in pixels, pixelsum, to estimate dose. Foldback values as well as track count may set to drive area switch limits. A value of 8.0×10^{6} was selected for foldback value and a track count of 2000 for pixelimit 1 .

This corresponds roughly to 250 mRem. With the additional track density as well as the increased track size, area read switching occurred at a lower level, approximately 250 mRem. While most routine dosimeters are typically below 250 mRem, we felt it desirable to raise the area read switch limit as high as possible without losing sensitivity.

To this end, we reduced lamp read brightness, see Chart 4 below, to a setting of 50, read a series of detectors exposed to 200 mRem of ^{252}Cf bare and a series of background detectors.
The process was repeated at lamp brightness increments of 25. Pixelsum was plotted along with tracks per cm squared. It can be seen that by decreasing lamp brightness, neutron induced tracks per cm^2 per given area read may be maximized while decreasing the relative non neutron induced background signal, thereby widening response range without resorting to inter range correction factors or sacrificing lower limit of detection.

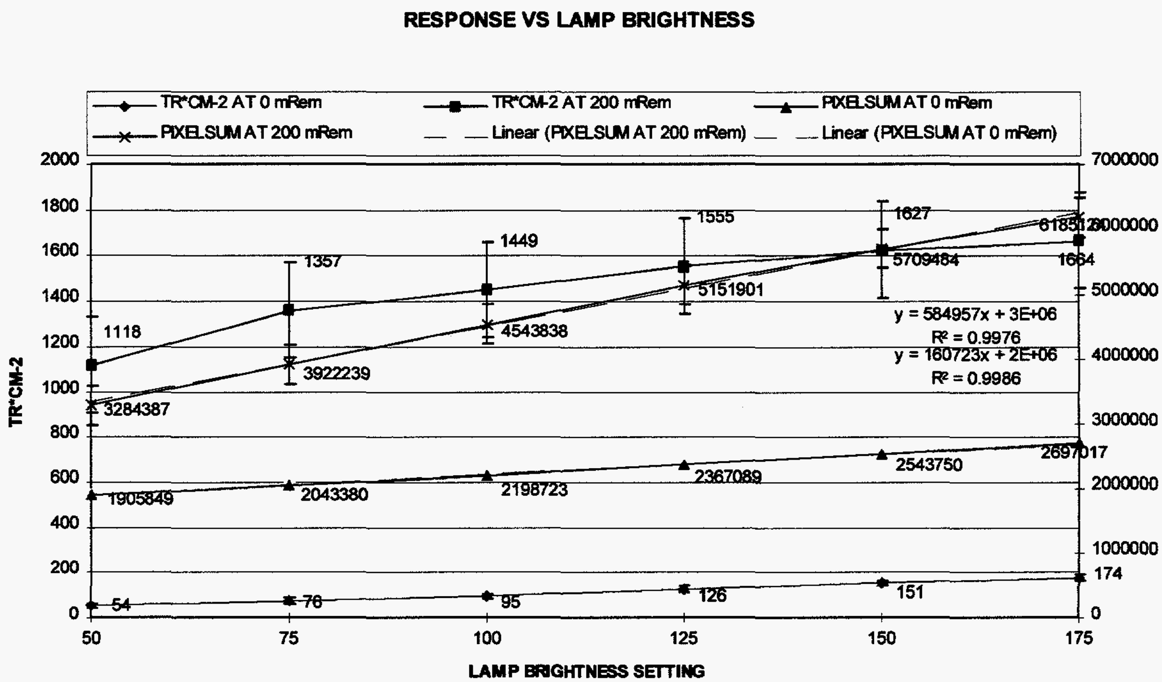


Chart 4

In Chart 5a, next page, the July 1997 audit detectors were read using a 15 hour etch with a one hour pre etch. We have opted, based on the results shown in Chart 4, for a lamp brightness setting of 75. With this setting in place for issue dosimeters, our background values have shown 41 ± 7 tracks per square centimeter and response range is between 10 and 300 mRem. Area read switching does not occur until exposures approach 350 mRem.

At the current time we are looking at developing a background build up function instead of using a fixed value, assigned based on QC Receipt testing. Preliminary testing indicates a function of Bg = 37 + .295d where 37 tracks is background upon receipt and d = the number of days between receipt and etching. In the near future we hope to identify a function to define material sensitivity fade.

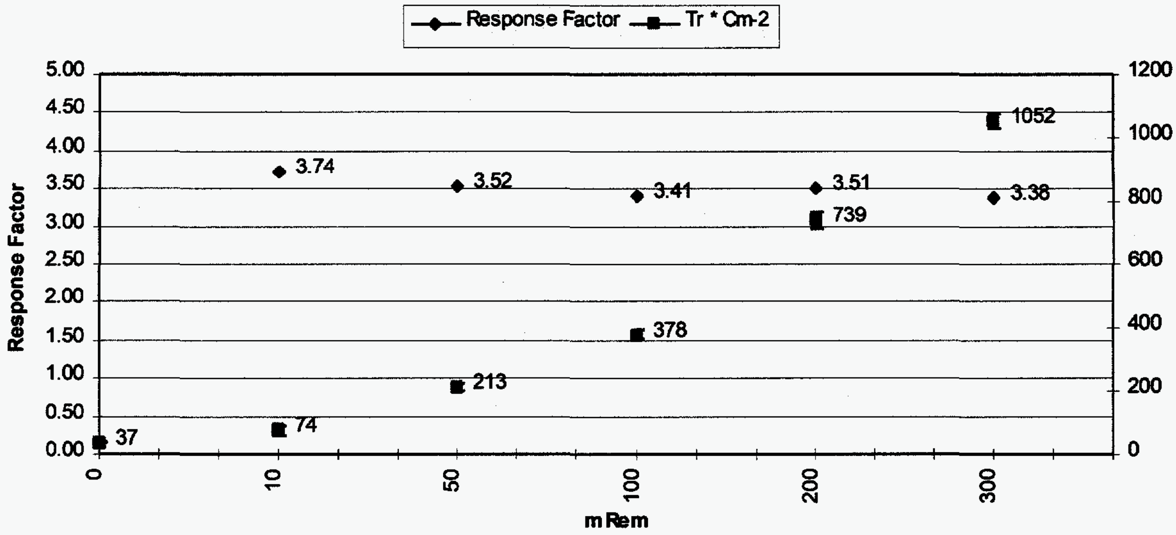


Chart 5a

In the event exposures exceed foldback values, pixelsum may be correlated to exposure. Pixelsum can be demonstrated to be linear with respect to exposure, see chart 5b, with the upper measureable limit influenced by lamp brightness. The lower the lamp brightness, the higher the exposure that may be estimated prior to the pixelsum curve losing lineararity.

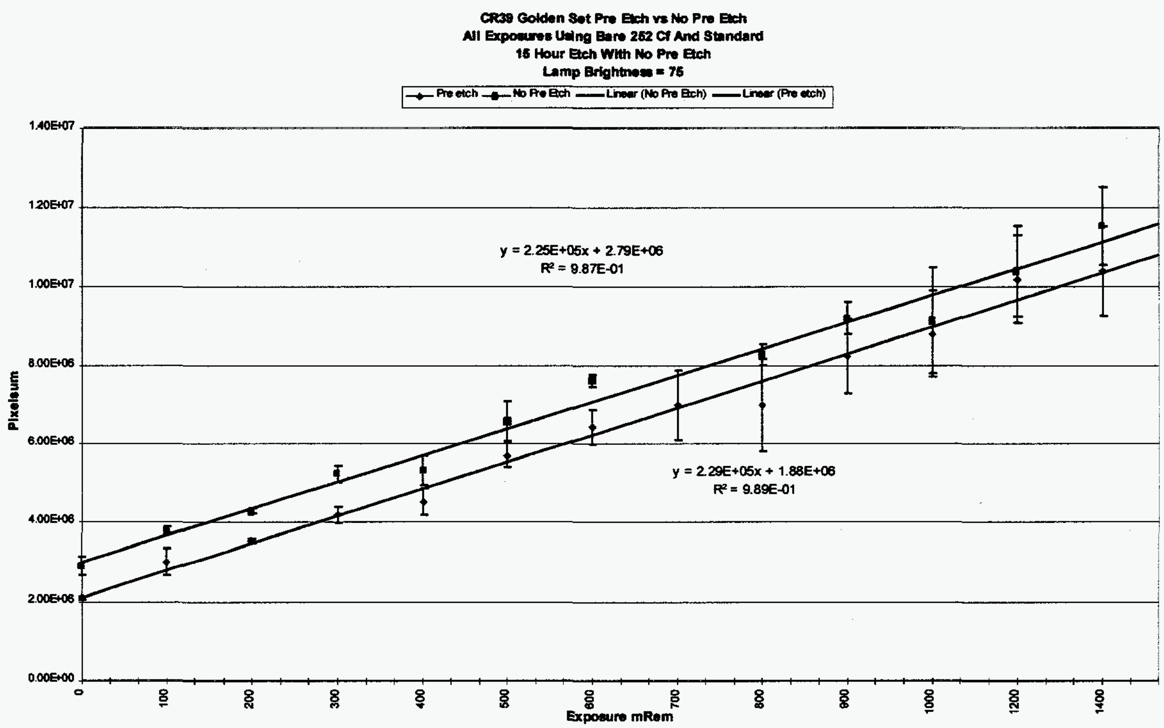


Chart 5b

The inter range correction coefficients were calculated, (Chart 6) and applied to the July 1997 audit dosimeters, whose results appear in Chart 7 below.

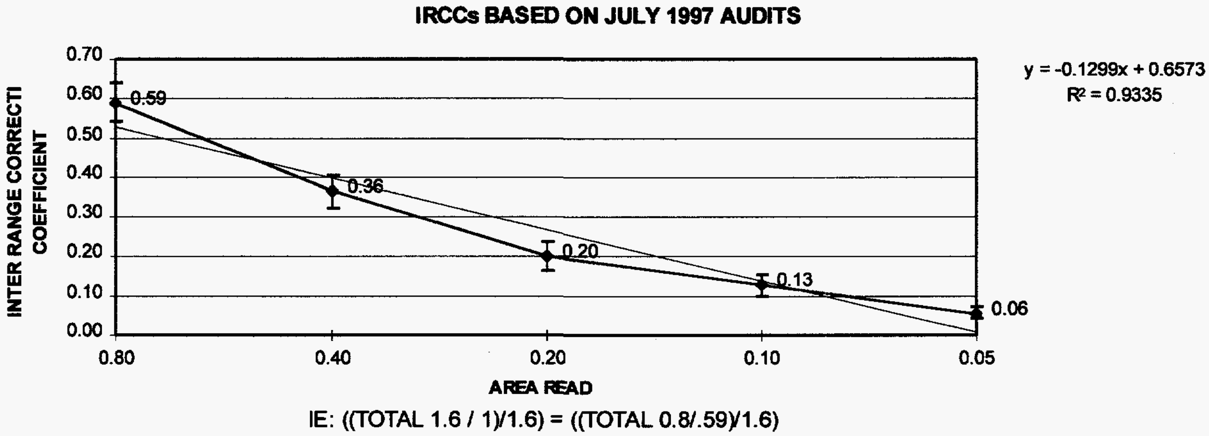


Chart 6

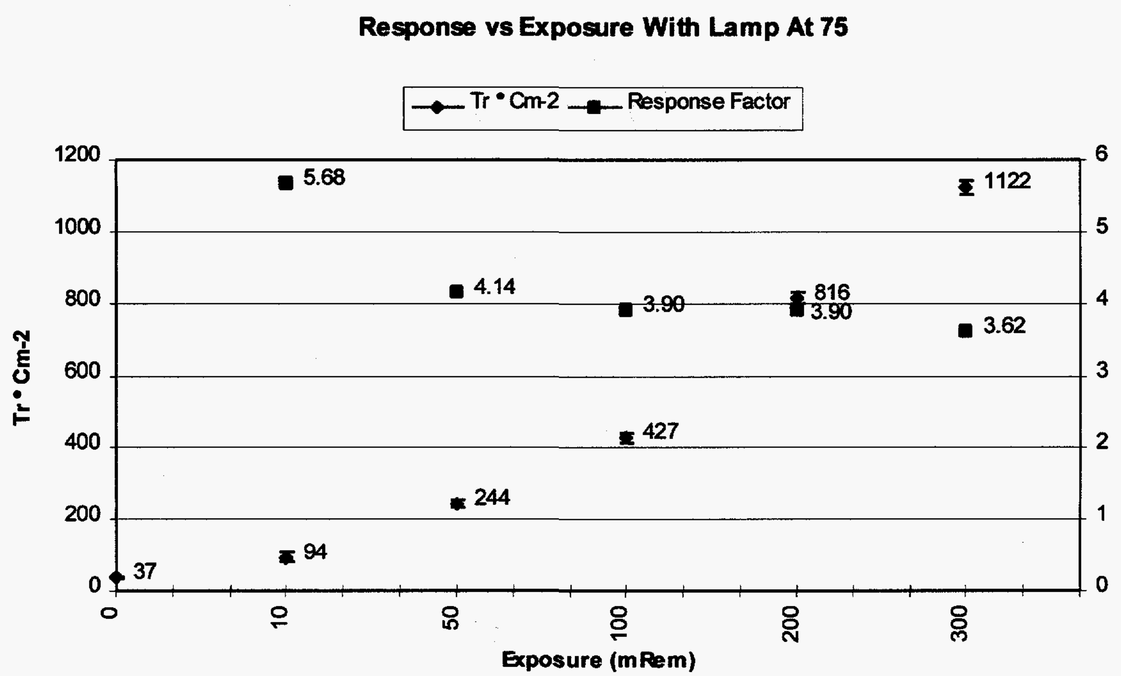


Chart 7

QC testing of new detector material is carried out on all new material received. QC testing typically consists of selecting 6% of detectors, from each sheet and batch, exposing half of them to 200 mRem of bare ^{252} Cf and using the other half as unexposed background controls (see Chart 8). Those detectors responding within two standard deviations of the mean are accepted for use as well as background detectors showing less than 150 tr * cm^{-2}.

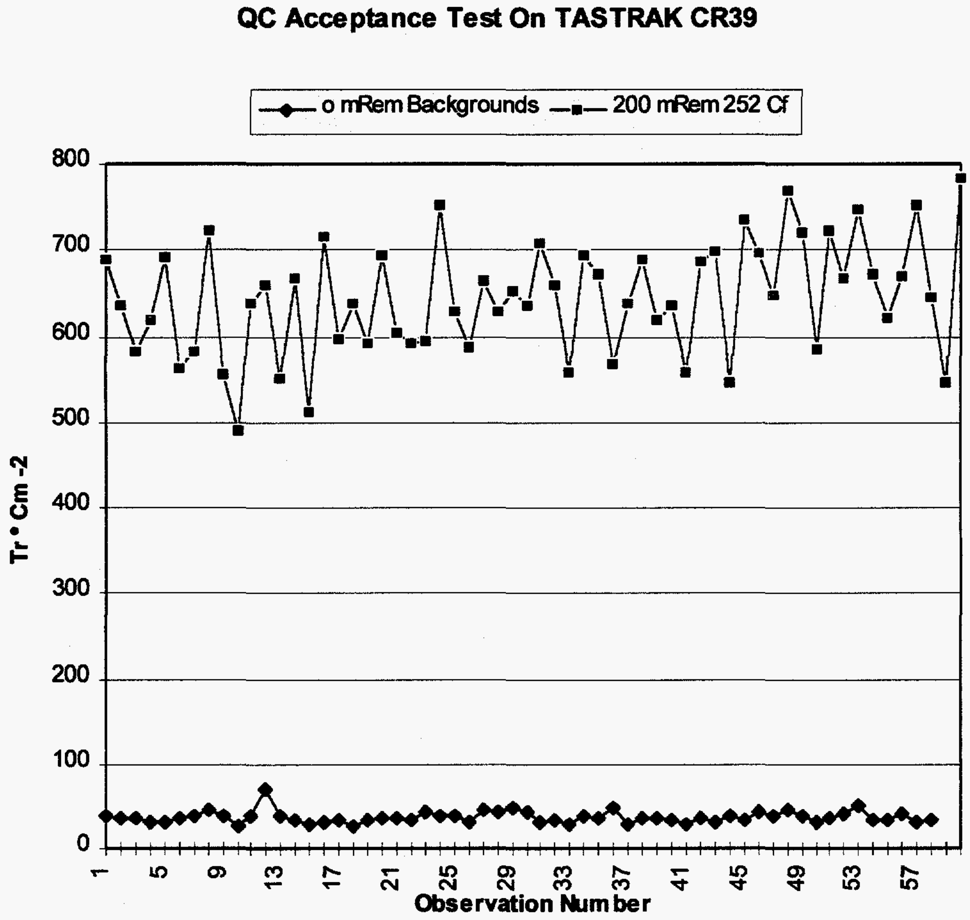


Chart 8

The fluctuation that can be observed in the 200 mRem exposures , above, is partially due to exposure geometry. A full explanation of this is given on the following page under Chart 9.

Additional QC checks are run on the reader itself as well as with each individual etch run and read. Prior to reading issue detectors, a set of five background and five exposed control detectors are read and the data entered into an Excel spreadsheet where the tr * cm^{-2} are converted to mRem. The mean and standard deviation are taken. The response of the means must be within 2 sigma of the mean response from the control foil population. The standard deviations are compared using a chi squared statistic corresponding to 95% CI, ie: $X^2 = (9.488/4)^{.5} = 1.54$.

Each Autoscan 60 carrousel has the capacity to hold 20 dosimeters, assuming 3 detectors per dosimeter. In place of the first and last three dosimeter detectors, we place three background and three 200 mRem control detectors respectively. This is done prior to etching and serves as a QC check on the etch process as well as the reading of the detectors.

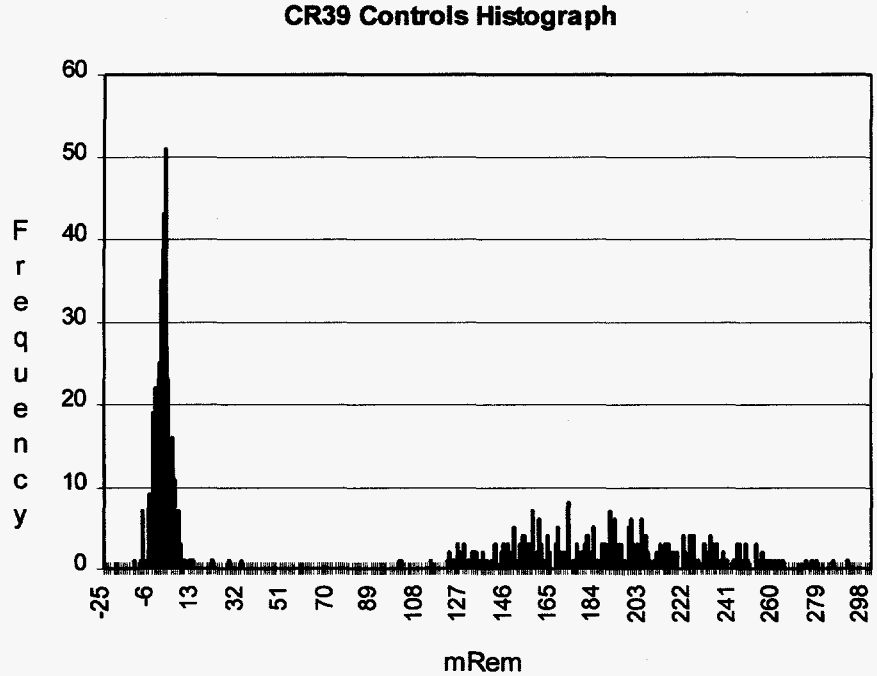


Chart 9

In chart 9, above, a histograph of the controls shows a large dispersion in the 200 mRem population. To investigate the cause for this, nine dosimeters, each containing three detectors were arranged on a 40cm x 40 cm x 15cm lucite phantom with the detector positions recorded. After exposure, the detector doses were calculated and compared to a model based on detector area normal to the neutron source. The detector angles are in fact responsible for the large dispersion in the 200 mRem exposures.

Personnel dosimeters are issued on a quarterly cycle. Following are the results of the first three month issue period (See Chart 10) using the new material received June 17, 1997. The dose distribution looks as may be expected and less than one percent of exposures were found to be in excess of 20 mRem, (LLD @ 95% CI). It is suspected that LLD may be lowered as we gain more experience.

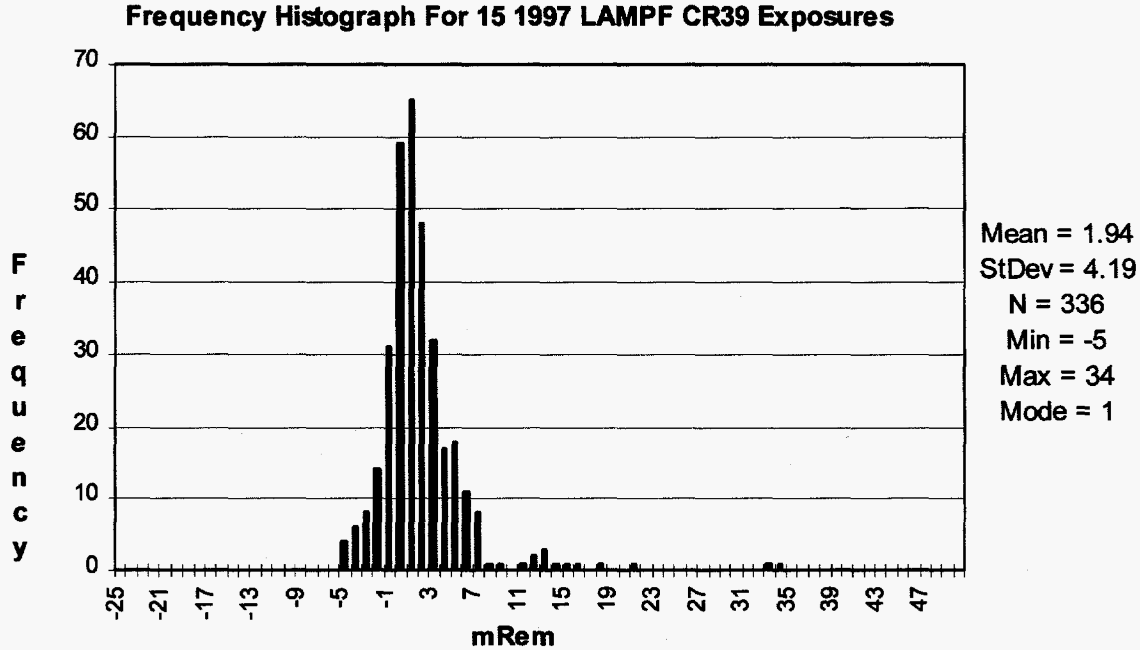


Chart 10