

## Personnel Neutron Dosimetry Using Electrochemically Etched CR-39 Foils\*

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## INTRODUCTION AND SUMMARY

We have developed a personnel neutron dosimetry system based on the electrochemical etching of CR-39 plastic at elevated temperatures. This dosimetry system is superior to any neutron dosimeter used previously. The doses obtained using this dosimeter system are more accurate than those obtained using other dosimetry systems, especially when varied neutron spectra are encountered. This CR-39 dosimetry system does not have the severe energy dependence that exists with albedo neutron dosimeters or the fading and reading problems encountered with NTA film.

The dosimetry system employs an electrochemical etch procedure that can be used to process large numbers of CR-39 dosimeters. The etch procedure is suitable for operations where the number of personnel requires that many CR-39 dosimeters be processed. Our experience shows that one full-time technician can etch and evaluate 2,000 foils per month. To improve the energy dependence, we adopted the hot (60°C) low-frequency (60 Hz) etch procedure recommended by Tommasino (1983). The energy response to neutrons is fairly flat from about 80 keV to 3.5 MeV, but drops by about a factor of three in the 13-16 MeV range. The sensitivity of the dosimetry system is about 7 tracks/cm<sup>2</sup>/mrem, with a background equivalent to about 8 mrem for new CR-39 foils. The limit of sensitivity is approximately 10 mrem. The dosimeter has

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a significant variation in directional dependence, dropping to about 20% at 90°. We etch the foils in two stages to improve the track quality and the precision of the track counting. The two-stage etch procedure also reduces the background counts caused by imperfections, scratches, or dirt on the foils. We have been using this dosimeter and procedure for personnel neutron dosimetry at the Lawrence Livermore National Laboratory for more than 18 months.

#### ETCH CHAMBERS

The etch chambers we are using are of the Homann-type and can handle 8 or 24 foils simultaneously. The chambers are not commercially available as yet and must be made in a good quality machine shop. Drawings of both the 8- and the 24-cell chambers are included in Appendix A of this report. The 24-cell etch chamber is the most useful and several are required for a dosimetry program. The 8-cell chambers are used only if small or odd numbers of foils are to be etched and one or two of the 8-cell chambers is adequate for most dosimetry facilities. The cells are made of Lucite and have one liquid electrode and one aluminum-plate electrode. These chambers are easy to handle, and have been used daily for more than a year with no difficulties. Several of these chambers can be processed at the same time with a single power supply. These chambers were designed to be used in a 60°C oven. Other etch chambers have been designed, but because of the excellent results we have had with these chambers, we strongly recommend that you use them during your initial trial period. The only maintenance that is required of the etch chambers is replacing the O-rings when they have become permanently distorted (after one month of daily use) and replacing the rubber Silastic seal around the stainless steel electrode when a leak develops (about every two weeks).

#### ETCHING PROCEDURE

The etching parameters that we are using are shown in Table 1. We are presently investigating the effect of etching high voltage on the energy dependence of the CR-39 foils and when this study is complete a change in the etching high voltage may be recommended if an improved energy dependence is

obtained. We have investigated the effect that changes in the other etching parameters have on the results, and we do not expect to make changes in these parameters. The etch chambers, loaded with the CR-39 foils, are placed overnight (or over a weekend) in an oven maintained at 60°C. The following morning, 60°C KOH is added to the chambers, which starts the first etch step. The power supply is manufactured by Homann-Bell (1985), and is programmed using a HP-41CX calculator to provide selected voltages, frequencies, and times. The second step, which we call blow up, is very important because it increases the size of the tracks that exist at the end of the first etch step, making them much larger and reasonably uniform in size. This greatly improves the precision that can be attained with the optical counting system because the tracks are large compared to the imperfections, dirt, scratches, etc. often present on the CR-39 foils. The background from the foils is reduced by using a higher track-size threshold on the optical reader, which allows us to discriminate against most of these small imperfections. The precision of the results are typically within  $\pm 1\%$  for repeated readings of the same foil.

When the etch chambers and foils are left in the oven over the weekend, the number of tracks/cm<sup>2</sup>/mrem on the foils is 5% lower than the number of tracks usually obtained with the overnight etch. For long weekends, the decrease is larger--being about 3% to 4% for each additional day.

The track density on the foil is determined by scanning the foils with the an optical bacterial colony counter (Biotran). Six fields of view, 3 x 3 mm, are counted by starting at the top-center of the etched area on the foil and moving down the center of the foil. If any of the fields have a peculiar appearance such as a scratch, ring of tracks, dirt or other abnormalities, that field of view is avoided and another selected, usually by moving sideways. There are about 13 possible fields in the etched area; we read only 6, which we feel is adequate to attain reasonable counting statistics.

A copy of the "Operating Procedures for Electrochemical Etching of CR-39" is given in Appendix B of this report. It describes the steps we use in the etching and reading of the foils.

## NEUTRON ENERGY RESPONSE

Low-frequency electrochemical etching at elevated temperatures produces a different energy dependence than that obtained with high frequency or chemical etching of CR-39 foils. Figure 1 shows the energy response we obtained in two separate studies. The upper curve was obtained using monoenergetic neutrons from the Tandem Van de Graaff accelerator at the Los Alamos National Laboratory, and the lower curve was obtained using an accelerator at the Battelle Northwest Laboratory. The slight difference in these curves is caused in part by a change in the etching procedures that occurred between these studies. The upper curve was obtained using our present etching techniques and is the curve that should be used. It indicates that neutrons with energies between about 80 keV and 3.5 MeV are detected with a relative flat energy dependence.

We are presently investigating the effect that changes in the etching high voltage and etching time have on the energy dependence. A flat energy dependence over the largest possible range of energies is desired. If our studies show that a different etching high voltage or etching time would give a better energy response curve, we will recommend changes in the etching parameters in a later report.

CR-39 does not respond to thermal neutrons. Using our etching parameters, there is also no response to neutrons between 0.01 and about 0.05 MeV, and a lower than desired response to neutrons with energies in the 13-16 MeV region (see Fig 1). We are now studying the effect of changes in the etching parameters on the high-energy response. It may be that changes in the etching voltage can also be used to improve that response.

The energy dependence of CR-39 foils using our etching procedures is superior to that obtainable with albedo neutron dosimeters or from chemical etching of CR-39. In Table 2, we show the results obtained using a  $^{252}\text{Cf}$  source moderated in the polyethylene, water,  $\text{D}_2\text{O}$ , and aluminum spheres that are used for studies in our calibration facility. The results indicate that the energy dependence of CR-39 using our etching techniques is sufficiently

flat to give CR-39 readings within a few percent of the actual dose equivalence, except for low readings from the large D<sub>2</sub>O moderators. This under-response is caused by the thermal neutron contribution to the dose equivalent, which is significant for the larger D<sub>2</sub>O spheres and is not detected by CR-39. For comparison, the calibration factors for albedo neutron dosimeters used with these moderated fields range over one decade. The important finding of this study is that the CR-39 response is nearly constant for the large water and polyethylene moderated neutrons. This is important since these are similar to the materials used in neutron shielding and storage containers. Therefore, the response of the CR-39 will be correct for leakage neutrons through shielding or from a storage container and still be correct for the unmoderated source when it is removed from the shield or container.

The CR-39 dosimeter will be under-respond to the neutron spectrum present inside the containment of a power reactor. In addition to the thermal neutron component, which is around 4 to 8 percent, about half of the remaining neutron dose equivalent is delivered by neutrons having energies of less than 100 keV (based on multisphere neutron instrument measurements). The CR-39 responds to only half the actual neutron dose equivalent.

If the CR-39 dosimeters are calibrated using a <sup>252</sup>Cf source and the person is exposed to a PuBe source, the results will be low by about 40% because many of the PuBe neutrons have energies above 4 MeV, where the response of the CR-39 falls off.

The personnel dosimeter system should be calibrated with CR-39 foils located in the badge. Figure 1 shows that the personnel badge decreases the response of the foils to neutrons. The dosimeters must also be on a phantom, since the backscatter increases the response of the CR-39 foils by about 7%. The foils must also be properly loaded in the holder with the side to be etched in the same position as it is in the personnel dosimeters. The dose used for calibration should be slightly below the point where the foils become nonlinear (about 3000 tracks/cm<sup>2</sup> which is about 400 mrem for our foils and etching procedures).

## EFFECT OF VARIOUS ETCHING PARAMETERS CHANGES

We have determined the effect of changes in the etching parameters on the dosimeter response. Figures 2 and 3 show the track density as a function of high voltage. For foils exposed to neutrons, Fig. 2 shows that the track density is not a strong function of the high voltage. Recently we have found that part of this decrease is caused by changes in the energy dependence at different etching voltages. The effect of high voltage on the background, shown in Fig. 3, is significant. The background track density increases exponentially as the etching high voltage is increased. We are presently etching at 3000 V but are investigating the effect that a change to a lower voltages would have on the background track density. Unfortunately, decreasing the high voltage may result in an unacceptable change in the energy dependence.

When the high voltage used in the first etching step is changed, it is necessary also to change the second-stage (blow up) etching time to keep the proper track size. Figure 4 shows the blow up time required to keep the proper track size.

Oven temperature is very important in the etching procedure. Before we recognized its importance, we would place the etch chambers in the oven and immediately start the etching process using room temperature KOH. Later, we measured the temperature of the KOH inside the cells. The results are shown in Fig. 5. The 24-cell etch chamber did not reach the temperature of the oven even after six hours. The 8-cell chamber reached equilibrium temperature after three hours. The result was that the 8-cell chamber gave us more and larger tracks than the 24-cell chamber. The room temperature also varied, which caused the initial temperature of the etch cycle to vary. To solve these problems, we now place the etch chambers, loaded with the foils, in the oven the night before the etch is to be performed. The KOH is also placed in the oven in a plastic squeeze bottle. The next morning the KOH is added to the etch chamber to start the first etch step.

Figure 6 shows the effect of changes in the oven temperature on the track density. Small changes in oven temperature are important and must be

avoided. At an oven temperature around 60°C, a 1°C change in oven temperature causes about a 3.5% change in the track density. An oven with a digital temperature controller ( $\pm 0.1^\circ\text{C}$ ) is required. If several etch chambers are to be used simultaneously, an oven with forced-air circulation is required to keep the temperature throughout the oven uniform. The temperature sensor should be located in the oven near the etch chambers.

If oven temperatures other than 60°C are to be used, the blow up time must be adjusted. Figure 7 shows the blow up time required as a function of the oven temperature.

The track density is a linear function of the total etching time; i.e., the sum of the first- and second-stage etching time. The time required for the second stage (blow up) as a function of the first-stage etching time is shown in Fig. 8. As the first-stage etching time is reduced, the blow up time must be increased to keep the track size correct.

The track density is affected by variations in the thickness of the foils. Thicker foils reduce the field strength, which results in smaller tracks. The opposite effect occurs for thin foils, and the tracks become too large. The track density varies by about 1% per mil of foil thickness. This can normally be ignored because the thickness variation is small in the sheets of CR-39 recently being received from the supplier ( $\pm 2$  mils). If the average thickness of the CR-39 sheets varies more than 3 mils from the specified 25 mils, the blow up time will have to be adjusted or the size of the tracks will be too large for thin sheets and too small for thick sheets.

Track density is also affected by the KOH normality. We use a hydrometer to determine the normality (at a specific gravity of 1.276). We have investigated the effect of changes in the KOH normality on the results and have found no significant difference in the track density for normalities between 6 and 7. Therefore, we recommend the KOH normality be  $6.5 \pm 0.25$ . We have found that repeated use of the KOH causes a reduction in track density by about 1% for each use. This change is not detectable with the hydrometer and is therefore caused by changes in the KOH that do not affect the density. We recommend that each batch of KOH be used a maximum of five times.

We use a 15-min post etch which makes the tracks rounder and therefore less ragged and therefore, more suitable for automatic counting. However, this has no effect on the evaluation of track density. At 30 min, however, the track quality begins to change, and therefore the foils should be removed from the etch chamber within 25 min after the completion of the etch cycle. An important feature of the post etch is that the cells do not have to be removed from the oven precisely at the end of the second etch cycle. This gives the technician flexibility in removing the cells, and if several cells are being used, they can be removed at various times within the 25-minute period.

Standard foils, exposed to 400 mrem of  $^{252}\text{Cf}$ , are included in each etch chamber to ensure that the foils are properly etched. If only one etch chamber is being used we include four standard foils in the chamber. If more than one chamber is being used, we reduce the number of standard foils to three and use the average of all the standard foils etched that day for the calibration.

#### PERFORMANCE OF THE DOSIMETER SYSTEM

##### Linearity

The linearity of the dosimetry system is shown in Figs. 9 and 10. Figure 9 was obtained when the neutron sensitivity of our etching procedures was only about 4 tracks/cm<sup>2</sup>/mrem. The curve is linear out to about 1.5 rem (4000 tracks/cm<sup>2</sup>). The curve shown in Fig. 10 was obtained when our sensitivity was about 8 tracks/cm<sup>2</sup>/mrem and is linear only to about 450 mrem but also to about 4000 tracks/cm<sup>2</sup>. These curves show that the linearity is a function of the number of tracks on the foil and not the neutron dose equivalent. Our reader is linear to about 4000 tracks/cm<sup>2</sup>. The dose equivalent corresponding to 4000 tracks/cm<sup>2</sup> depends on the efficiency of the system at the time the etch is performed. The linearity can be extended to higher dose equivalence by reducing the etch time or changing some of the other etching parameters, which will produce fewer tracks/mrem. The foil results can also be corrected for nonlinearity by using the curves shown in Figs. 9 and 10 if the track density is less than about 15,000 tracks/cm<sup>2</sup>.

If the CR-39 foils are exposed to high neutron dose equivalents, it may be necessary to reduce the first-stage etching times to less than five hours. The etching time can be reduced to keep the track density within the linear region of the dose response curve or to obtain a track density that falls within the range of the curve, enabling a correction for nonlinearity to be made. We have used first-stage etching times as short as 1 hour but the track quality is poor. At two hours, the track quality is adequate, and therefore etching times down to two hours are acceptable. If the track density is still too large, a lower etching temperature may also be used. Extending the etching time beyond five hours requires a reduction in the blow up time to the point where the track quality is poor (large variation in track sizes occur when inadequate blow up time is used), and therefore etching longer than five hours is not recommended.

#### Sensitivity

Our present CR-39 dosimeter and etching procedure result in a sensitivity of about 7.5 tracks/cm<sup>2</sup>/mrem. The background on new foils varies but is around 60 tracks/cm<sup>2</sup> which is equivalent to about 8 mrem. This gives us a limit of sensitivity ( $\alpha = \beta = 0.05$ , see Appendix 4) of approximately 10 mrem for a single CR-39 foil. The standard deviation of the track count for foils exposed to 400 mrem (bare <sup>252</sup>Cf source) varies with the individual sheets but is between 3 and 5%. For background foils, it is around 30%.

In our recently completed one-year study of storage techniques, we inadvertently placed some of the foils in an area that had a previously undetected neutron field of about 6 mrem/yr above background. This was easily detected, but required that a number of foils be evaluated and the results averaged. One can determine neutron fields smaller than the environmental neutron background (about 6 to 8 mrem/yr at LLNL) if a number of foils are used.

#### Directional dependence.

The directional dependence of CR-39 is a strong function of the angle of incidence of the neutrons, as shown in Fig. 11. As the angle of incident

neutrons approaches 90 degrees, the response of the CR-39 foil drops rapidly. This is caused by the recoil protons entering the foil at a high angle and the protons path is nearly parallel to the surface of the foil. When the foil is etched, no track develops. The curve shown in Fig. 11 is different from the curve obtained with collimated protons (Cross and Ing 1983). The neutrons used to obtain Fig. 11 were from a small uncollimated  $^{252}\text{Cf}$  source. Many of the neutrons have been scattered, and in addition the protons produced by the neutrons are emitted at various angles. The effect is a rounding of the angular response curve from that obtained by Cross and Ing using protons.

Figure 11 shows that the track density is less for neutrons entering the foils through the back ( $180^\circ$ ), than for those entering through the front ( $0^\circ$ ). The CR-39 sheets have a 5-mil-thick polyethylene protective covering. Polyethylene has a greater hydrogen density than CR-39, and therefore the proton production, ( $n, p$ ) with hydrogen, is greater. Consequently, the side of the foil facing the source has a higher track density than the back side by about 25%. By etching the side of the foil next to the person, we can use this effect to improve the directional dependence.

The directional response obtained from CR-39 foils in a personnel badge located on a phantom is shown in Fig. 12. The lower solid curve, obtained by etching the side of the foil next to the wearer, is the best directional dependence attainable at present. This directional dependence decreases to about 20% at  $90^\circ$ , but this is in reasonable agreement with the ~30% decrease in the 1 cm depth dose delivered to tissue exposed at  $90^\circ$  (see NCRP Report #38).

We apply a correction factor to partially compensate for the error that one would have in the dosimetry results caused by the directional dependence. We selected as our calibration point a value 20% lower than the results obtained from a face-on ( $0^\circ$ ) exposure. This means that if a person were exposed face-on to the source, his actual dose equivalent could be underestimated by 20%. But for exposures at the higher angles of incidence, the error in underestimating his dose equivalent would be reduced. The disadvantage of etching the back of the foil is that the sensitivity on the

back side of the foils is about 30% less than the value on the front. This reduces the sensitivity of the dosimetry system from about 8 to 5 tracks/cm<sup>2</sup>/mrem. The background track density on the CR-39 foil is not changed, and the effective dose equivalence of the background is increased. We feel the improvement in the directional dependence justifies the changes in the sensitivity and background and is a reasonable health physics approach to the directional dependence problem.

Efforts to improve the directional response of CR-39 have been only marginally successful. We attempted to improve the directional dependence by bending or arching the foils. The results indicated a slightly improved directional response that was too small to justify the time and effort involved in bending the foils.

#### DESCRIPTION AND EVALUATION OF THE LLNL BADGE

At LLNL, we are using the Panasonic TLD Dosimeter. The TLDs are placed in a plastic badge holder that was designed at LLNL. This holder contains the beta and low-energy x-ray shield and has slots into which the components of the nuclear accident dosimeter can be placed. At the time this holder was designed, CR-39 foils were not being used and no provision was made to include them in the holder. To hold the CR-39 foils, we glued a 1/8-in.-thick piece of plastic to the back of the badge holder. An end-mill was used to cut though the back of the badge holder and into the plastic to form a recess. We insert three of the CR-39 foils into this recess with the side to be etched (low background side) next to the wearer. The CR-39 foils are well protected and are not exposed to ambient light (which is essential). When the foils are placed in the badge, a felt tip pen is used to mark each of the foils with a number that identifies the wearer and the month the badge was issued.

When the badges are returned at the end of the month one of the foils is etched. If the reading from that foil is greater than 6 mrem above the background, the other two foils are etched on following days. The average reading of the three foils is used to calculate the neutron dose equivalence the person is assigned. If the track density of the first foil is less than 6 mrem, the remaining two foils are not etched. With this procedure, we are

able to avoid reading the second and third foils for individuals who did not receive any significant neutron exposure. We can determine most of the neutron exposures above 10 mrem, missing only an occasional exposure (caused by the statistics of the foils at low doses and of the background).

#### CR-39 FOILS

##### Foil Stability and Fading

All of the CR-39 foils that we have used have been manufactured by American Acrylics. They are "dosimetry grade" CR-39 made of high-purity monomer (about 94% pure) purchased from Pittsburg Plate Glass (PPG). Standard grades of CR-39 cannot be used in personnel neutron dosimetry. The CR-39 sheets are 25 mils thick and vary little in thickness over the sheet ( $\pm 2$  mil). The sheets are covered on both side by American Acrylics with a nominal 5 mil thickness of polyethylene, which is required to protect the foil from radon alpha particles and to protect the foils from abrasion. At present nothing is added to the monomer. Previously an antioxidant was added, but our studies indicate it has no effect on the short-or long-term quality of the CR-39.

Our sheets are laser cut by Applied Fusion Inc. of San Leandro, California. The foil size we use was selected to allow the foil to be placed inside the Hankins type albedo neutron dosimeter. We have discontinued the use of these albedo dosimeters but the foil size has not been changed because it is convenient to handle and we get over 500 foils from each CR-39 sheet. Larger foils are being considered by others to permit the use of a bar code to identify the foils.

The CR-39 foils have a reasonably low background on the side of the sheet that was on the top of the mold during casting. The background track density on the back of the sheet is about 10 to 15 times higher than on the front. We still do not know the reason for this difference. In any event, this requires that the side of the sheet that was on the top of the mold be marked to assure the proper orientation of the foils when they are laser cut. We do this by drawing lines with a felt tip pen at about a 45 degree angle on the

polyethylene covering the sheets. When the foils are used, to assure that the foils have the same side up, the lines on the foils are all oriented in the same direction. In some cases, the side of the sheet that was on the top of the mold during casting has been mislabeled, and a test run with the foils must be performed before they are used. Another marking method we have used is to cut off one corner of each foil at a 45 degree angle. The advantage of this method is that the foils are permanently marked to indicate proper orientation.

We performed a seven-month fading study which indicated that little fading of latent tracks had occurred (Hankins et al., 1985). A six-month environmental study indicated that about 18% fading does occur if the foils are kept at higher temperatures (40°C) (Hankins et al., 1986). A recently completed one-year study of storage conditions indicates that when the foils are protected from light, little if any fading or change in sensitivity is occurring, and the background increases at a rate consistent with the environmental neutron background. Foils used in this study that were not protected from ambient light faded, lost sensitivity, and suffered background increase to the point where the foils were useless. We conclude from the above studies that fading, changes in sensitivity, or increases in the foil background are not a problem if the foils are protected from light. It has been reported, however, that CR-39 sheets made by an English supplier lose sensitivity at a reported rate of 8% per month but show no fading of the latent tracks (Harrison 1986). We are interested in understanding the difference between the English CR-39 and that from American Acrylics so that we can avoid having the U.S. produced sheets suddenly develop the same problem.

The CR-39 foils are stable for routine use as personnel neutron dosimeters. Our studies have shown that as long as the CR-39 foils are protected from light and exceptionally high temperatures, they are not damaged and the dose equivalent information is retained. The CR-39 foils are damaged, however, by UV light or prolonged exposure to high temperatures. The mechanism for damage is not known, but may be associated with the adhesive that is on the protective polyethylene cover placed on the CR-39. When the

foils are exposed to UV or ambient room light for several months, the surfaces of the etched foils have many small dots or globs of some material on them. These dots apparently cause tracks to appear on the foil. As these dots become larger from additional exposure to light or heat, the number of background tracks increases. The background tracks are more dense in areas on the foils where these small dots are more dense. We believe that these dots are the adhesive on the polyethylene which has deteriorated and remains on the foils when the polyethylene is removed. If care is taken to protect the foils from light or excessive heat, the foils are useful for at least one year. The CR-39 foils could be issued for a six-month exchange period. It should be remembered, however, that the background track density on the foils will be increased by the environmental neutron background. To keep the background of the CR-39 foils as small as possible, only fresh foils should be issued.

#### Storage of New Foils.

The foils must be stored in the dark. Earlier we stored the material in a refrigerator or freezer, but the results from our one-year study of storage techniques indicate that reduced temperatures are not necessary. Storage in a refrigerator provides a very convenient method of storage, guaranteeing a dark storage environment. We will continue to use our refrigerator for storage of new foils.

#### COST AND EQUIPMENT

A list of the items required to establish a CR-39 dosimetry system is given in Appendix C. An estimate of the cost of the major items is also given. Not included is the cost of a bar coding system and computer used for dosimeter identification and the dosimetry records.

#### ADDITIONAL STUDIES.

We are presently studying the effect of changes in the high voltage on the energy dependence of the CR-39. Voltages from 0 to 3500 are being studied. This study includes foils that were exposed to various monoenergetic neutrons on and off a phantom and from the front and back of the foil. The

results from these foils will be used to determine if the backscatter from the phantoms remains the same at all neutron energies and if the ratio for foils exposed from the back or the front remains the same at all neutron energies. Some of the exposed foils will be etched using a first-stage etching time of two hours, and the results will be compared to those from a five-hour etch to determine if etch time has any effect on the energy dependence. These results should be available within the next few months.

In future studies, we will expose CR-39 foils to 14 MeV neutrons and determine what effect changes in the high voltage have on the energy response of CR-39 to these neutrons. We will include foils to determine the effect of phantoms and exposures of the foils from the front and back.

Although the CR-39 foils being produced now are adequate for our present needs, we must have foils with lower backgrounds and possibly higher sensitivities if the quality factors are increased or lower neutron dose equivalents are to be measured. Some improvements in CR-39 may be possible through the use of high-purity monomers or different molding techniques. Copolymers, made by mixing CR-39 monomer and other materials, have been produced that etch much more rapidly than CR-39. When these materials become available, we will investigate their potential as personnel dosimeters.

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Table 1. Recommended etching parameters

	<u>Etching</u>	<u>Blow up</u>	<u>Post Etch</u>
High Voltage	3000 V	3000 V	0
Frequency	60 Hz	2.0 KHz	0
Temperature	60°C*	60°C	60°C
Time	5 hours	23 min**	about 15 min
KOH normality	6.5 N	6.5 N	6.5 N

\* The etch chambers and KOH must be left in the oven overnight (or weekend)

\*\* No adjustment for foil thickness is required if the foils are within 3 mil of 25 mils.

Table 2. Spectrum dependency of CR-39 for moderated  $^{252}\text{Cf}$  neutrons.

$^{252}\text{Cf}$ Moderator	<u>Percent deviation in track density per rem</u>	
	For neutron energies > 0.1 MeV	For all neutron energies
None	0	0
2 cm poly	+9.5	+8.4
5 cm	+5.0	+1.5
10 cm	-5.7	-5.1
25 cm $\text{H}_2\text{O}$	-4.7	-6.7
5 cm $\text{D}_2\text{O}$	+4.1	+7.8
10 cm	+0.5	-5.3
15 cm	+0.1	-14.1
25 cm	-2.6	-31.9
20 cm Al	-3.6	+2.1

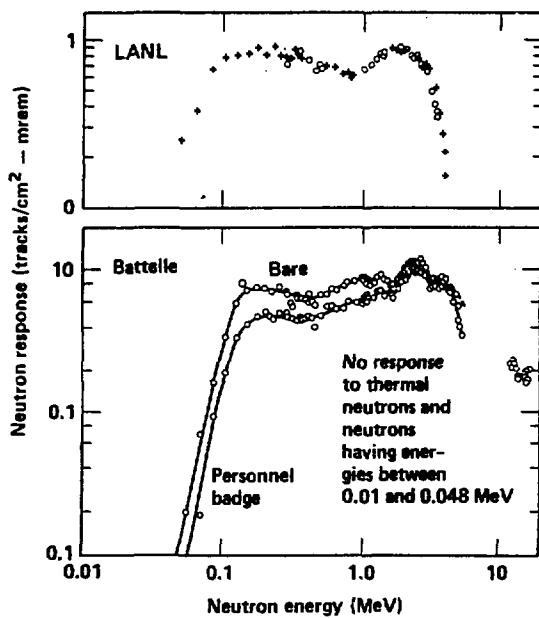


Figure 1. Energy dependence of CR-39 personnel neutron dosimeters.

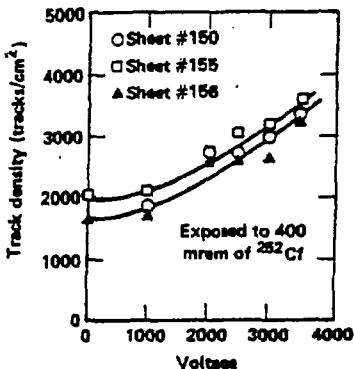


Figure 2. Track density as a function of etching high voltage.

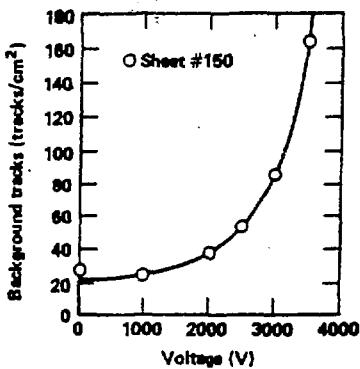


Figure 3. Background track density as a function of etching high voltage.

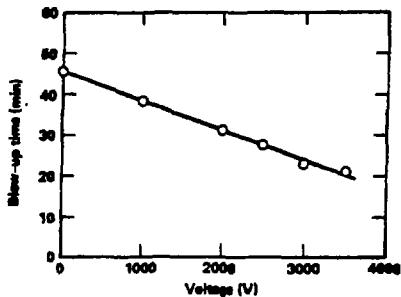


Figure 4. Blow up time as a function of etching voltage.

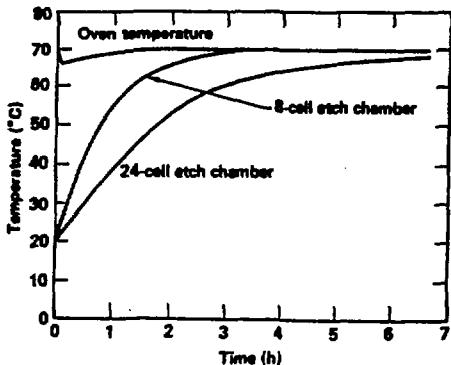


Figure 5. Temperature inside the 8- and 24-cell etch chambers as a function of time.

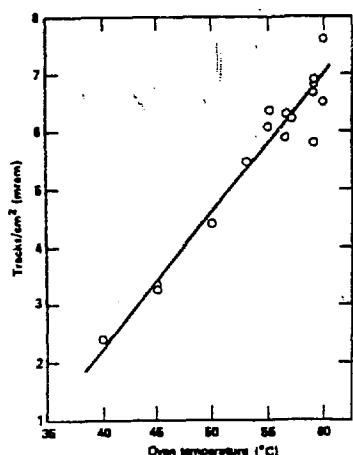


Figure 6. Track density as a function of oven temperature (5-hour etching cycles using 6.5 N KOH).

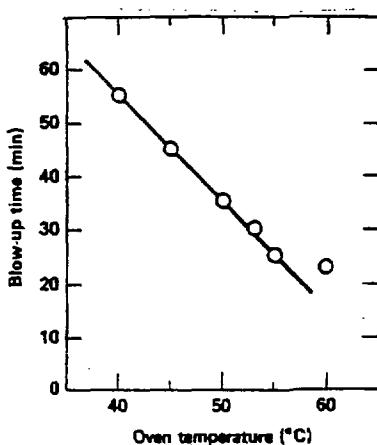


Figure 7. Blow up time as a function of oven temperature (5-hour etch cycles using 6.5N KOH).

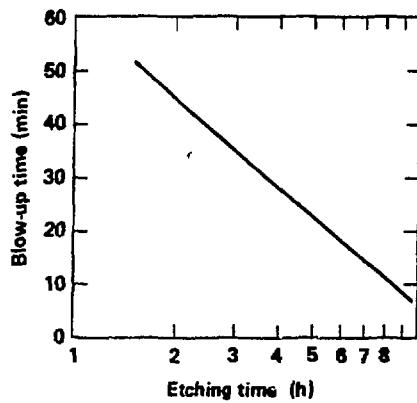


Figure 8. Blow up time as a function of etching time (6.5 N KOH).

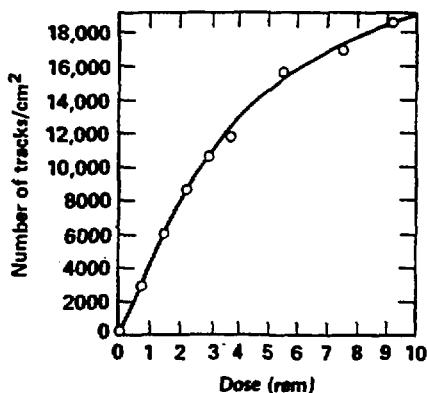


Figure 9. Linearity of the CR-39 foils produced in June 1984.

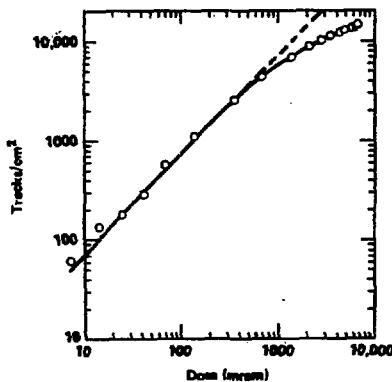


Figure 10. Linearity of the CR-39 foils produced in October 1986.

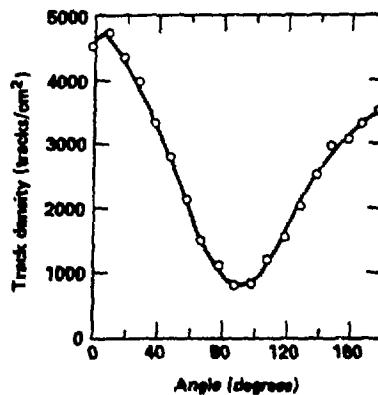


Figure 11. Directional response of CR-39 foils in air.

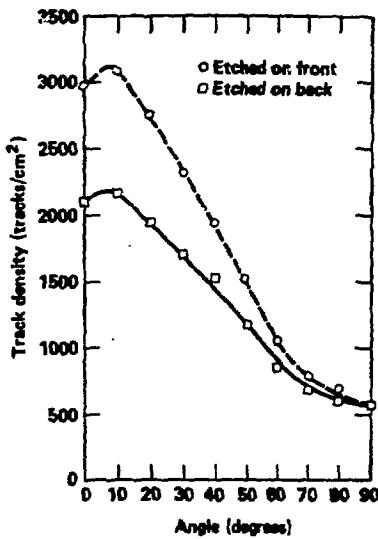


Figure 12. Directional response of CR-39 in a personnel badge on a phantom.

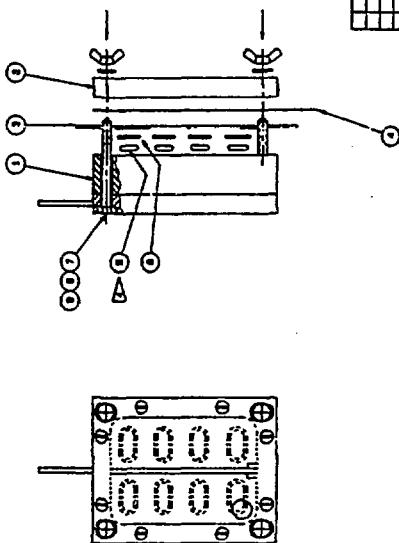
#### Appendix A

This appendix contains engineering drawings for the 8-cell and 24-cell Homann-type electrochemical etch chambers.

**NOTE** **DATA** **CHARTERED** **EXPIRED**

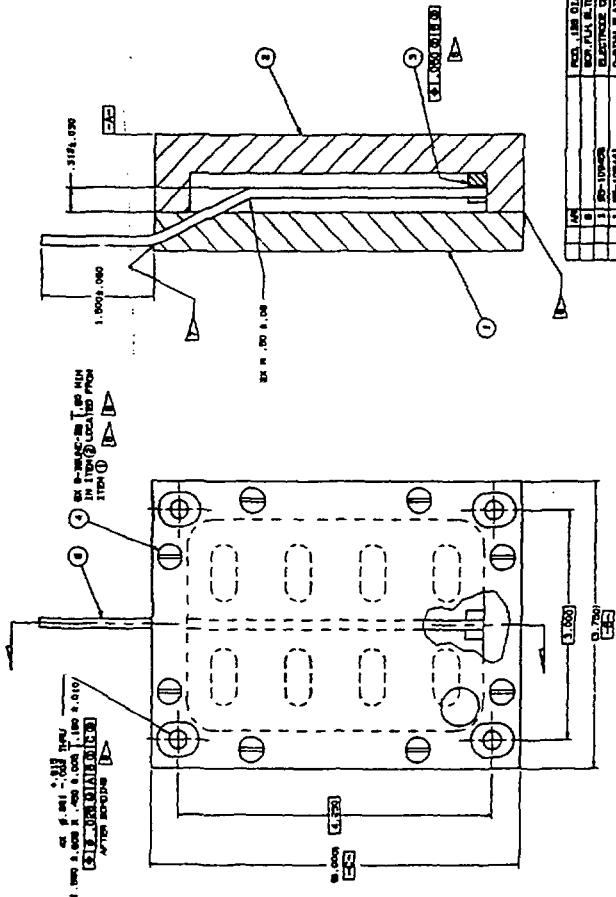
1. **QUADRANTONE AND TORONTO** **9/18**  
**AM 9/16-1972.**
2. **AMERICAN TRUST FOR AMERICA** **1-1978.**
3. **ALL DOCUMENTS ARE IN BOUND.**

**□** **LETTERS AND TO BE PLACED**  
**IN ENVELOPE ON SHELF**



NOTA  
UNA OTRAVIADA MECANICA

1. DISMINUIR ANG. TORCIMIENTO PER  
UNA 1/2 A 1/4 DEG.
2. SURFACE TESTORE PER AÑOS 84.1-1916.
3. AL DIMINUIR ANG. TORCIMIENTO.
4. LIVELLO EN ESTADO.
5. APRETAR BOLAS .100 MM.
6. BOLAS TYP 3 A TYP 6  
AL TORNAR SIN TORNILLAR DEDALERA.
7. BOLAS TYP 6 A TYP 4 CON  
SISTEMA DE TORNILLADO.
8. BOLAS TYP 1 A TYP 4 CON  
ESTRUCTURA DEDALERA. LINEA TYP 4  
DEBE DE QUEDAR SUELTA Y SIN TENSION.



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1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100

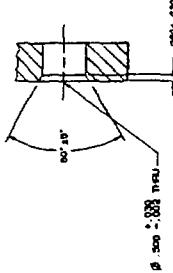
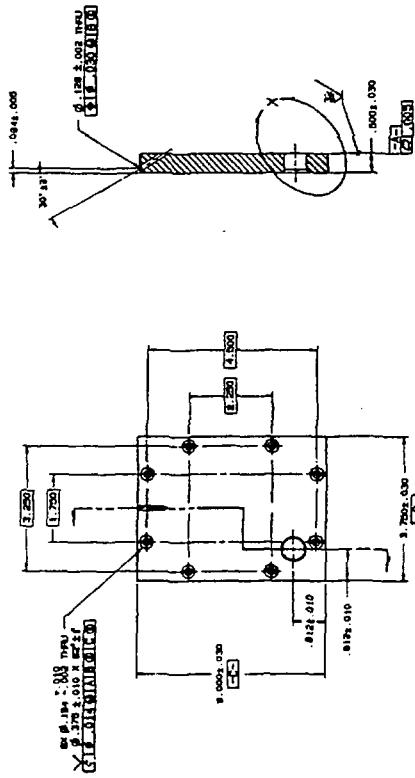


**NOTES**  
UNLESS OTHERWISE SPECIFIED:  
1. DIMENSION AND TOEBRACING PER  
AMHS Y14.3M-1982.  
2. SURFACE FINISH PER AMHS 844.1-1978.  
3. ALL DIMENSIONS ARE IN INCHES.

4. STOCK 1

5. BREAK EDGES IN .020 MAX.  
ON CHAMFER

6. POLISH OPTICALLY CLEAR.

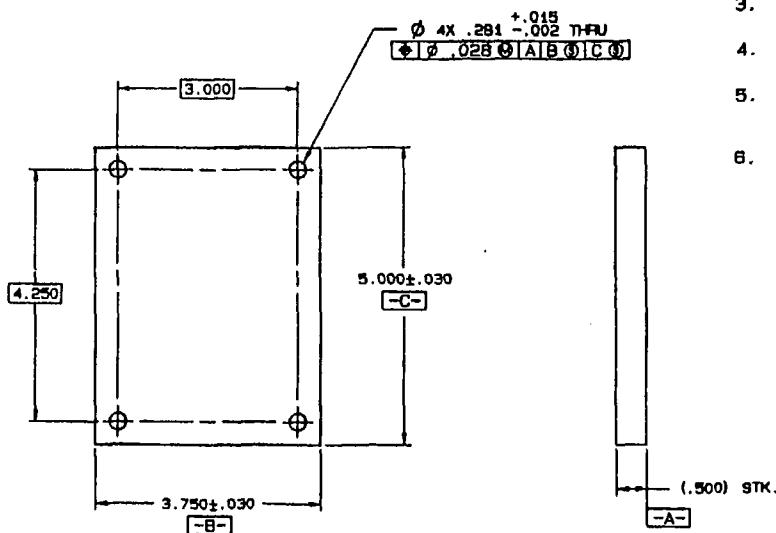


DETAIL "X"

## • NOTES

UNLESS OTHERWISE SPECIFIED

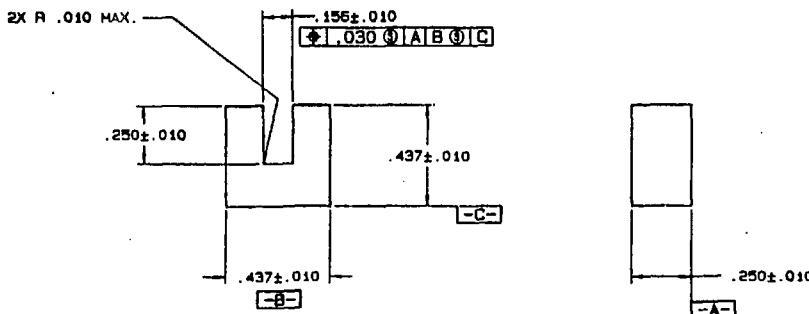
1. DIMENSIONING AND TOLERANCING PER ANSI Y14.5M-1982.
2. SURFACE TEXTURE PER ANSI B46.1-1978.
3. ALL DIMENSIONS ARE IN INCHES
4.  $\frac{3}{8}$  & STOCK
5. BREAK EDGES R .020 MAX.  
OR CHAMFER
6. TAG PART WITH DWG. NO.



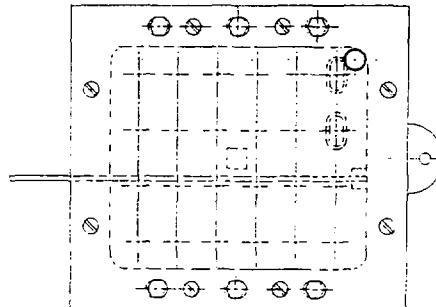
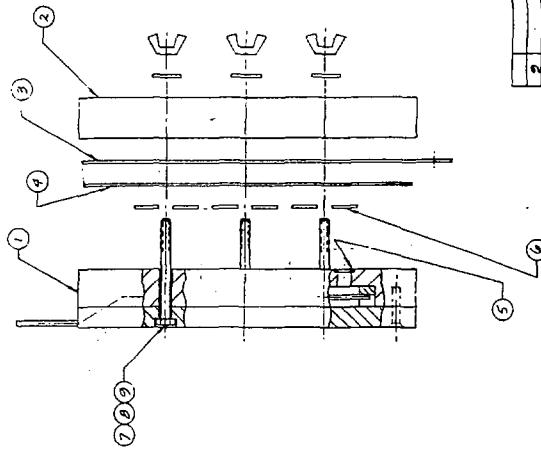
## NOTES

UNLESS OTHERWISE SPECIFIED:

1. DIMENSIONING AND TOLERANCING PER ANSI Y14.5M-1982.
2. SURFACE TEXTURE PER ANSI B46.1-1978.
3. ALL DIMENSIONS ARE IN INCHES.
4.  $\frac{63}{3}$  OR STOCK.
5. BREAK EDGES .020 MAX.
6. TAG PART WITH DWG. NO.

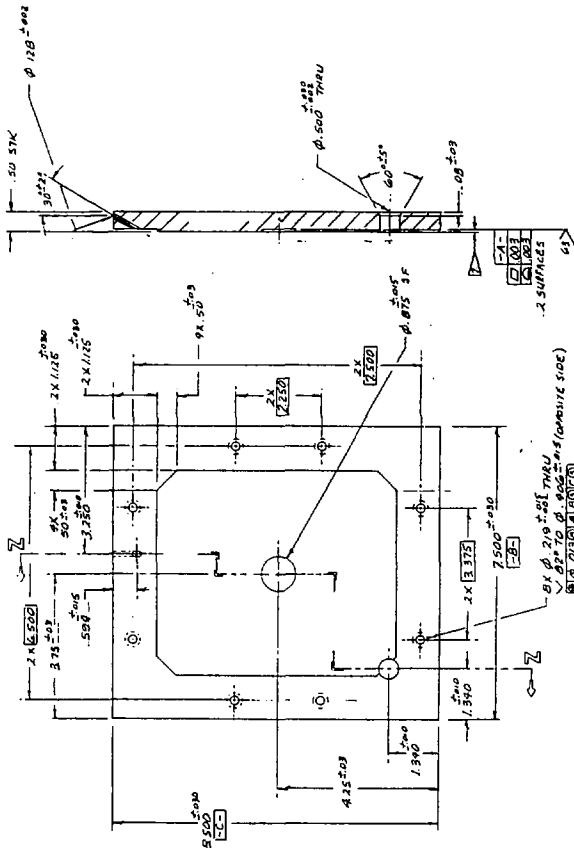


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	CHK	<i>[Signature]</i>	<i>[Signature]</i>	INFORMATION AND PROPERTY OF THE UNIVERSITY OF CALIFORNIA	HOMANN TYPE	
	APV	<i>[Signature]</i>	<i>[Signature]</i>	LAWRENCE LIVERMORE LABORATORY.	ELECTROCHMICAL ETCH CELL	
	CLASSIFIED BY			REPRODUCTION PROHIBITED WITHOUT	SUBASSEMBLY	
	TYPE			PERMISSION OF THE MECHANICAL	DETAIL	
	DATE			ENGINEERING DEPARTMENT.	ELECTRODE CLIP	
 LAWRENCE LIVERMORE NATIONAL LABORATORY MECHANICAL ENGINEERING DEPT UNIVERSITY OF CALIFORNIA			DRAWING NO 85-109456 ACT 0774-00 DATE 10/10/85		COPY AAA 85-109456-00 SHEET 1 OF 1	
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NOTES-

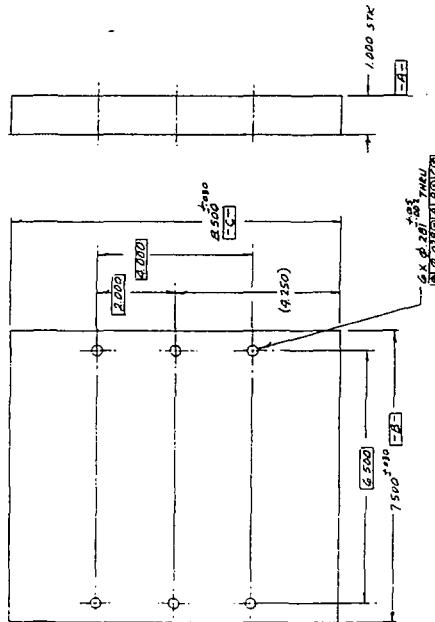
- 1. DUM & DOL FOR ANESTHESIA ENR-1982
- 2. SURFACE TEXTURE PER REQUEST
- 3. ALL DUMS ARE IN INCLINES.
- 4. ✓ NOTED.
- 5. REMOVE ALL WURS & REAR  
CARTERS OLD AND
- 6. CHECK SURFACES TO OPTICALLY
- 7. DUMMING WORKING TO REACH  
DUM FLATNESS



SECTION Z Z

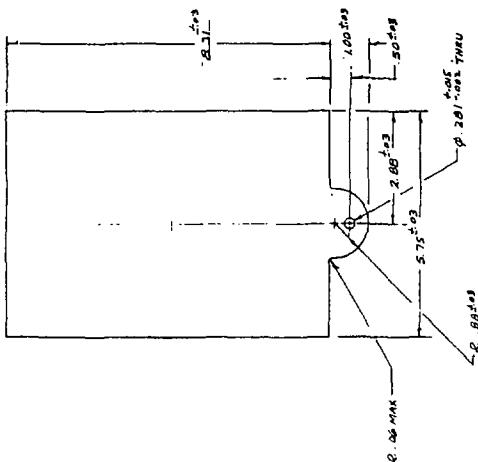
### NOTES—

- 1 DIM & TOL PER ASME Y14.5M-1992
- 2 SURFACE TEXTURE PER ASME Y14.6M-1992
- 3 ALL DIMENSIONS ARE IN INCHES
- 4 125<sup>+</sup> ALL MACHINED SURFACES
- 5 REMOVE ALL JUNKS & BREAK CORNERS. DEBUR.



### NOTES:-

1. DIM  $\delta$  TOL PER ANNOTATION.
2. SURFACE FEATURE PER ASSEMBLY.
3. ALL DIMS ARE IN INCHES.
4.  $\frac{1}{16}$  ALL MACHINED SURFACES.
5. REMOVE ALL BURRS & BREAK CORNERS ON MAX. DIAM.
6. THE PART WITH DING NO.



NOTES -

1. DRY VOL PER ANSIV 54-1992
2. SURFACE TEXTURE PERANSIV 54-1992
3. ALL BIMS ARE IN INCHES
4. ALL MACHINED SURFACES
5. ~~PERMIT~~ SQUARES FOR BREAK CORNERS

→ BOND ITEM 3 TO ITEM 1  
SIGHT WITH ENTHALICLORIDE

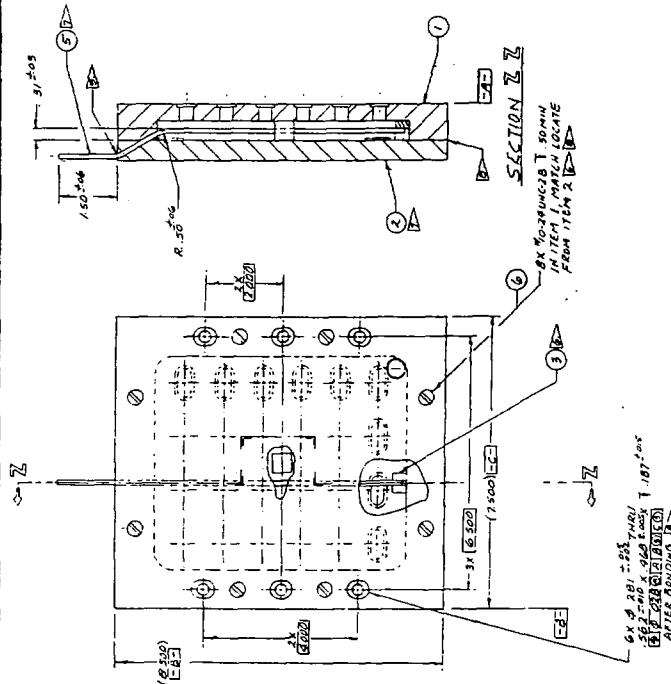
→ BOND ITEM 3 TO ITEM 2 WITH  
COPOLY DIBACRIDE NO. B

→ BOND ITEM 2 TO ITEM 1 WITH  
ENTHALICLORIDE NO. 16476 G.  
(A PLATE) TO CLAMP PLATES TOGETHER.

→ USE DRY CARBON 270-732  
ADHESIVE SCARF 75 SCALE 1270-570

6	See <u>Page 2</u> <u>Line 14</u> <u>1/1/1920</u>	Classification
7	See <u>Page 2</u> <u>Line 15</u>	
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## Appendix B

### OPERATING PROCEDURES FOR ELECTROCHEMICAL ETCHING OF CR-39

#### LOADING OF ETCH CHAMBERS

1. Check the oven temperature, and adjust if necessary.
2. Enter information relating to the foils and etching parameters in the daily log book and on the counting form or the computer.
  - a. Experiment # (assign consecutive identification numbers for each etch)
  - b. Etch chamber identification
  - c. Counting date
  - d. KOH normality
  - e. Biotran reader settings
  - f. Temperature of oven
  - g. First etching step parameter
    1. High voltage
    2. Frequency (Hz)
    3. Etching time
  - h. Second etching step (blow up) parameters
    1. High voltage
    2. Frequency
    3. Blow up time
  - i. CR-39 sheet number
  - j. Letter or number to be used on each foil
  - k. Identify exposure to foils (worn by, placed at, background, calibration dose, etc.)
3. Remove the protective polyethylene cover from the top of the foil using a scalpel or knife. Write the experiment number at the top of the foil and the identification letter or number at the bottom of the foil. These must be written small enough to avoid the area of the foil that will be etched later.

4. Remove the protective polyethylene covering from back of foil.
5. Position CR-39 foils face down on the O-ring around the cell opening.  
Place foils in order on the etch chamber, using the letter and number on the foil.
6. The chamber is connected to the house vacuum using the chamber's vent hole. Accurately position the foils over the opening and turn on the vacuum. It may be necessary to press the foils down onto the O-rings using the Lucite pressure plate before a good seal can be obtained. The vacuum will hold the foils securely while the etch chamber is being assembled.
7. Place one of the 10-ml pieces of polyethylene over the foils.
8. Place one of the aluminum plates on top of the polyethylene. This aluminum plate is the second electrode.
9. Place the Lucite pressure plate on top of the chamber and secure it to the chamber body with the bolts and wing nuts. The wing nuts should be securely fastened by using fingers only.
10. Shut off the vacuum and remove the hose. Plug the blood pressure gauge into the vent hole to test the chamber for leaks. The pressure will remain constant if a good seal has been made and the chamber does not leak.
11. Place the chambers and the KOH bottle in the oven and leave there overnight. Remove the chambers and the KOH from oven the next morning and immediately partially fill (~ 3/4 full) the chamber with KOH through the vent hole. Secure a rubber stopper (with a 1/16-in. hole) in the vent hole. This prevents spillage when the cell is tipped but still allows air to vent through the small hole.

12. Attach the high-voltage leads to the electrodes (rod and aluminum plate) using the alligator clamps or telephone jacks. If a second chamber is being used, additional high-voltage leads or jumper cables will be required.
13. Place the chamber in a plastic photo tray with the vent hole positioned at the front of the oven. The oven racks must slant slightly to the rear of the oven. This prevents the KOH from being forced out through the small hole in the stopper if the KOH and air expands.
14. Check the power supply parameters that are in the HP-41 calculator by pressing the yellow button and then the "edit" button. The calculator will display the parameters in sequence each time the R/S button is pressed. If a change is required, enter the new value in the calculator and press the R/S button. To check your input, repeat the readout procedure for the etch parameter. To start the etch, press the yellow key and then the RUN key.
15. Check the oven temperature and high voltage periodically during the etching cycles.
16. Leave the chambers in the oven for a 15-minute post etch following the completion of the etch cycles.

#### Unloading

Empty the chamber of KOH by removing the stopper and pouring the KOH back into the bottle. Use a funnel to prevent excessive spillage. Rinse the chamber with tap water at least twice. Then take it apart, rinse all the parts, and leave them to air dry. Soak and rinse the foils in distilled water for about 5 min. Then remove the foils and blot them dry using paper towels. Before reading them, wipe them with lens paper.

Counting Procedures

Calibrate the Biotran counter each day using the standard foil. Position the foil so that the same 3 x 3 mm area on the foil is counted. When it is properly adjusted, the Biotran counter will indicate the same number of tracks each day. Record the Biotran reader settings. Obtain six counts from each foil to be read, usually starting at the top of the foil and progressing down the middle of the foil. At each position, adjust the microscope focus until the highest number is obtained. If abnormalities such as scratches, odd shapes of tracks, etc. are noticed on the foil, select another position on the foil and obtain a count. The etched area of the foil will allow a maximum of about 13 of the 3 x 3 mm areas to be counted.

## Appendix C

### Equipment Required

Oven with temperature controller	\$ 2,000.00
Refrigerator	500.00
Power supply	6,700.00
Biotran counter	10,000.00
Microscope	3,000.00
Computer	?
Etching chambers	\$500 to \$800.00 each
KOH pellets	
Plastic squeeze bottles for KOH	
Funnel to return KOH to squeeze bottle	
Paper towels for drying foils	
Storage containers for CR-39	
Scalpel	
Aluminum plate (1/16 in. thickness) electrode	
Polyethylene sheets (10 mil) between electrode and foils	
Permanent ink pens to mark foils for I.D.	
Lens paper to wipe foils	
Photo trays to protect oven from spills	
Beakers for rinsing foils and etch chambers	
Graduated cylinders for mixing KOH	
Blood pressure gauge for leak testing	
Plastic gloves	
Notebook	
Glassine bags to store etched foils after reading	
Stapler to secure glassine bags	
Hydrometer to measure a specific gravity of 1.276	

## APPENDIX D

The distribution of CR-39 track events is well represented by the Normal distribution. Consequently, the limit of sensitivity, LOS, is defined as follows:

$$LOS \text{ (mrem)} = \frac{[k^2 + 2k[sdev(N_b)]]}{S} \quad \text{Eq. 1}$$

where:  $k = k_\alpha = k_\beta$

$k_\alpha$  = the abscissa of the standardized Normal distribution corresponding to the probability level,  $1-\alpha$ .

$k_\beta$  = the abscissa of the standardized Normal distribution corresponding to the probability level,  $1-\beta$ .

$sdev(N_b)$  = Standard deviation of the CR-39 foil background - track/cm<sup>2</sup>.

$S = \text{Sensitivity of the CR-39 foils} = \frac{\text{tracks}}{\text{cm}^2 - \text{mrem}}$ .

For our CR-39 dosimetry system,  $k = 1.645$  ( $\alpha = \beta = 0.05$ ), the standard deviation of the background is 20 tracks, and the sensitivity is 7 tracks/(cm<sup>2</sup>-mrem). Therefore, using the above equation,

$$LOS = \frac{[1.645^2 + 2(1.645)(20)]}{7}$$

$$LOS = 10 \text{ mrem.}$$

For an observed dose of 1.0 mrem, the probability of stating the dose is greater than background, when in fact it is background, is 5%. The probability of stating that the observed count is less than background, when in fact it is greater than background is also 5%.

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