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## Neutron Irradiations of Proton-Sensitive Track Etch Detectors: Results of the Joint European/USA/Canadian Irradiations

Organized by EURADOS-CENDOS (1986)

E. Piesch Hauptabteilung Sicherheit

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Kernforschungszentrum Karlsruhe

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## KERNFORSCHUNGSZENTRUM KARLSRUHE Hauptabteilung Sicherheit

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Edited by E. Piesch

Kernforschungszentrum Karlsruhe GmbH, Karlsruhe

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## Abstract

In 1986, the EURADOS-CENDOS group organized a joint neutron irradiation of proton-sensitive track etch detectors particularly CR-39 and CN-85. The purpose of this experiment was to investigate the energy response and the background track density of plastics under development for use as personnel neutron dosemeters.

Dosemeters from 13 laboratories in the European Communities, the USA and Canada were sent to five laboratories and irradiated with monoenergetic neutrons of 144 keV, 250 keV, 570 keV, 1.2 MeV, 5.3 MeV, 14.7 MeV, and with Cf-252 neutrons, in the dose equivalent range between 1.5 mSv and 4.2 mSv.

The report consists of short papers from each participant giving a description of the dosemeter used, the processing and evaluation method employed and a discussion of the measurement results.

Neutronenbestrahlung von protonen-empfindlichen Kernspurdetektoren: Ergebnisse der gemeinsamen Europäischen/USA/Kanadischen Bestrahlung 1986 organisiert von EURADOS-CENDOS

## Zusammenfassung

Die EURADOS-CENDOS-Gruppe organisierte 1986 eine gemeinsame Neutronenbestrahlung von protonen-empfindlichen Kernspur-Ätzdetektoren insbesondere CR-39 und CN-85. Zweck des Experiments war die Untersuchung der Energieabhängigkeit und der Untergrund-Spurendichte des für Neutronen-Personendosimeter verwendeten Plastikmaterials.

Dosimeter von 13 Laboratorien der Europäischen Gemeinschaft, aus den USA und Kanada wurden an 5 Bestrahlungslaboratorien verschickt und dort mit monoenergetischen Neutronen von 144 keV, 250 keV, 570 keV, 1,2 MeV, 5,3 MeV, 14,7 MeV und mit Cf-252-Neutronen im Äquivalentsdosisbereich zwischen 1,5 und 4,2 mSv bestrahlt.

Der Bericht besteht aus Kurzbeiträgen eines jeden Teilnehmers mit einer Beschreibung des verwendeten Dosimeters, der angewandten Entwicklungsund Auswertemethode und einer Diskussion der Meßergebnisse.

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## EURADOS-CENDOS/USA/Canadian Joint Neutron Irradiation 1986: Introduction

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In 1984, the CENDOS Ion Recoil Neutron Dosimetry Group arranged neutron irradiations of proton sensitive track detectors (primarily CR-39 and CN-85) where mainly representatives of this group participated. The purpose of this joint irradiation was to investigate the energy response of their personnel neutron dosemeters under development and to compare their results with the data of other workers in the field. The published results of this joint irradiation have been the basis of comparison for all laboratories working with CR-39 plastics.

Because of the progress in the last two years, the new EURADOS-CENDOS group decided at a meeting in Rome in 1985 to organize a new joint program of neutron irradiations<sup>1</sup>). Other groups in the USA and in Canada were invited to participate with their dosemeters and to provide neutron irradiations. In the period between both joint irradiations most groups applied and optimized the two-step electrochemical etching technique proposed by Tommasino et al.<sup>1</sup>). In addition, chemical etching and the use of other plastics such as CN-85 have been further developed.

For the Joint European/USA/Canadian Neutron Irradiation the participants prepared up to 18 single standard-sized irradiation cards 10 cm x 10 cm in size. Fig. 1 shows an arrangement of the dosemeter cards of the participating

 This work was performed by EURADOS-CENDOS, a co-operative Europen Research Project on the Collection and Evaluation of Neutron Dosimetry Data, and was sponsored by the Commission of European Communities, Directorate General for Science Research and Development, Radiation Protection Programe laboratories, which was kindly provided by PNL. These cards were irradiated free in air with neutrons incident normally. Irradiation cards and background cards were sent to the five irradiation laboratories. Lists of participants and laboratories are given in Tables 1 and 2, respectively.



# Fig. 1: Collection of irradiation cards for one exposure at PNL provided by courtesy of Dr. Hadlock

The laboratories at AERE Harwell (A), Pacific Northwest Laboratory Richland (B), PTB Braunschweig (C), GSF Neuherberg (D) and CRNL Chalk River (E) provided irradiations with monoenergetic neutrons using neutron energies of 144 keV (C), 250 keV (C), 750 keV (D), 1.2 MeV (B), 2.85 MeV (E), 5.3 MeV (D) and 14.7 MeV (E). Irradiations with Cf-252 neutrons were performed by all five laboratories. For combinations of track detector and boron radiator phantom irradiations were done also with thermal neutrons (A). The dosemeters were irradiated in November 1986.

The track detectors were evaluated in the laboratories in the first months of 1987. The irradiation laboratories provided the relevant data of neutron fluence and dose equivalent using conversion coefficients published by ICRP 21<sup>2</sup>). The neutron dose equivalent exposures were between 1.5 mSv and 4.2 mSv.

In their short papers presented in this report, the participants provided in standard tables for each neutron irradiation the results of track density, background track density, the net track density and the neutron response given by the ratio of net track density and the reference dose equivalent. The background track density often may vary between those detectors sent to the irradiation laboratories and those stored in the laboratory of the participant.

For this report each participant prepared a short paper which describes the dosemeter used consisting of track detector and radiator, the process of chemical or electrochemical etching or a combination of both, the evaluation technique employed and the results obtained in the irradiation experiment. The short discussion of the results is focused to the properties of energy dependence, the background track density and quality of the detector plastic, i.e. the homogeneity in background and sensitivity, within sheets of the same production and within the individual sheet.

The report presents the state of the art in 1986 in the development of personnel neutron dosemeters based on proton sensitive track etch detectors.

## **References:**

- (1) K.G. Harrison (Editor), Neutron irradiations of proton-sensitive track detectors: results of a joint irradiation organized by CENDOS, AERE R 11926 and CENDOS Report 1985-O2, 1985
- (2) ICRP Publication 21, Data for protection against ionizing radiation from external sources. Oxford, Pergamon Press, 1973

Table 1: List of participants

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Table 2:List of irradiation laboratories

M. Cosack, PTB Braunschweig, FRG W.G. Cross, CRNL Chalk River, Canada D.E. Hadlock, PNL Richland, USA K.G. Harrison, AERE Harwell, UK H. Schraube, GSF Neuherberg, UK

## EURADOS - CENDOS/USA/Canadian JOINT NEUTRON IRRADIATION 1986: RESULTS FROM KARLSRUHE NUCLEAR RESEARCH CENTRE

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## 1. Description of the dosemeter

Two CR 39 materials were used manufactured by the American Acrylics in the thickness of about 700  $\mu$ m, and by the Pershore Moulding Ltd. in thickness of about 500  $\mu$ m with 2 - 6 % IPP initiator concentration and 0.2 % DOP platicizer. After delivery from the manufacturer the plastics have been spread in a dry nitrogen atmosphere before use for the experiment in October 1986. After identifying one surface side the sheets have been cut by rase plate into detectors having a size of 25x25 mm<sup>2</sup> and marked. Each single CR 39 dosemeter was then covered from both sides by a polycarbonate foil (Makrofol E of 300  $\mu$ m thickness). The polycarbonate foil serves as a radiator and protects the dosemeter against dust and scratches. The dosemeter have been fixed matrix like between two plates of Lucite with the marked side on the front face.

## 2. Processing

In our etching procedure the KfK system for electrochemical etching (ECE) has been applied which is described elsewhere (1). 20 detectors have been etched simultaneously on the front surface only. The etching procedure using 6 NKOH at 60° C consists of a two-step ECE technique:

- at 1500 V with frequency of 100 Hz for 5 hours and followed continuously by etching
- at 1500 V with frquency of 2 kHz for 1 hour.

The CR 39 foils usually fixed in the set of etching cells of the system before etching were kept over night in a thermo-controlled oven maintained at 60° C. Immediately before etching the 60° C etching solution were filled into the etching cells. Due to the difference in the detector thickness the field strength

was about 23 keV x cm<sup>-1</sup> for American Acrylics and 30 keV x cm<sup>-1</sup> for Pershore Moulding (r.m.s.).

A detector area of 1.5 cm diam. were etched. The track density have been counted in the central area of 1.2 cm diam. using a microfiche reader - printer and magnification of 40 times.

#### 3. Discussion of results

The results obtained for the CR 39 detectors in contact with the 300 µm thick polycarbonate radiators after irradiations at various laboratories during the EURADOS - CENDOS Joint Intercomparison Experiment 1986 have been summarized in Table 1 and Table 2 for the CR 39 detectors produced by American Acrylics and Pershore Moulding Ltd., respectively. In Figure 1, the values of the dose equivalent response has been plotted against the neutron energy.

From the Tables 1 and 2 and the Figure 1, both CR 39 plastics seem to be comparable in sensitivity and energy dependence with small differences in the energy range below 0.5 MeV. Energy dependence still exists for high energy neutrons of 14 MeV with a drop in sensitivity by a factor of about 2 in the region between 5 MeV and 14 MeV. The registration efficiency of recoil protons is limited for high energies due to the angular distribution of recoil protons. This cut-off has been found at about 3 MeV for etching conditions which are similar to ours (2). Because of the decrease in the cross section for the elastic scattering of protons the decrease in response at higher neutron energies might be improved only slightly by optimization of etching conditions using suitable radiator/absorber combinations.

As it is shown in the fifth column that the background reference foils returned from the 5 irradiation laboratories are nearly similar but considerably higher by a factor of 2 for the American Acrylics plastic in comparison with the Pershore Moulding plastic, except the background foils from the PTB. It is worthwhile to mention that the background track density is significantly higher compared to the background track density of detectors stored in our laboratory which is usually around 60 tracks/cm<sup>2</sup>.

The dose equivalent response of CR 39 plastic of Pershore Moulding for Cf-252 neutrons have been found to be consistent for irradiations in the different laboratories showing, however, an uncertainty of the mean value within  $\pm$  15 % for American Acrylics and  $\pm$  21 % for Pershore Moulding which may be explained by the non-homogeneity of the plastic with respect to sensitivity and background. Commercially available plastics are still far from being standardized

and the quality of material varies between the sheets of one production and sometimes in the same sheet which seriously limits the application also in the case of rejecting low quality sheets. In recently delivered CR-39 the background track density were found to vary between 50 and 500 tracks/cm<sup>2</sup> within one sheet.

### Reference

- (1) Urban, M., Wicke, A. and Kiefer, H., Bestimmung der Strahlenbelastung der Bevölkerung durch Radon und dessen kurzlebige Zerfallsprodukte in Wohnhäusern und im Freien, Sept. 1985, Report KfK 3805
- (2) Cross W.G., Arneja A. and Ing. H., The response of electrochemically-etched CR-39 to protons of 10 keV to 3 MeV, Nuclear Tracks and Rad. Meas., Vol 12, p. 649-652, 1986



Fig. 1 Energy response of ECE etched CR 39 manufactured by American Acrylics and Pershore Moulding Ltd. with 0.3 mm polycarbonate as radiator

No of samples	Neutron f or energ E (Me	field (y <sup>1)</sup> V)	Reference dose equivalent H (mSv)	Track density N±σ <sub>n-1</sub> (cm <sup>-2</sup> )	Background tracks N <sub>0</sub> ±σ <sub>n-1</sub> (cm-2)	Net track density N±SEM (cm <sup>-2</sup> )	Response R±SEM (cm <sup>-2</sup> ·mSv <sup>-1</sup> )
2	0.144	(C)	2.32	2814.8 ± 159.4	331.1 ± 30.6	2483.7 ± 144.8	1070.6 ± 49.5
2	0.250	(C)	1.73	1960.3 ± 326.4	331.1 ± 30.6	1629.2 ± 231.9	941.7 ± 233.9
2	0.570	(D)	1.50	1123.4 ± 204.5	347.5 ± 25.0	775.8 ± 145.2	517.3 ± 96.8
2	1.200	(B)	2.09	1521.7 ± 160.0	330.7 ± 20.0	1191.0 ± 114.1	569.9 ± 54.4
2	2.850	(E)	2.05	1675.1 ± 38.1	344.4 ± 23.1	1330.7±31.5	649.1 ± 15.4
2	5.300	(D)	2.12	1403.4 ± 352	347.5 ± 25.0	1055.9 ± 249.6	498.0 ± 117.7
2	14.700	(E)	4.20	1272.8 ± 160.7	344.4 ± 23.1	928.4±114.8	221.1 ± 27.3
2	Cf-252	(A)	2.27	1368.3 ± 93.2	261.3 ± 130.1	1107.3 ± 113.1	487.8 <b>± 49.9</b>
2	Cf-252	(B)	2.02	1551.8 ± 81.3	330.7 ± 20.0	1221.1 ± 59.1	604.5 ± 29.3
2	Cf-252	(C)	2.87	1914.3 ± 170.1	331.1 ± 30.6	1583.2 ± 122.2	551.6±42.5
2	Cf-252	(D)	1.99	1329.4 ± 59.4	347.5 ± 25.0	981.9 ± 45.5	493.4 ± 22.9
2	Cf-252	(E)	2.03	1642.4 ± 104.4	344.4 ± 23.1	1298.0 ± 75.7	639.4 ± 37.2

Table 1 : Result of 1986 Joint Irradiation, CR 39 American Acrylics

<sup>1)</sup> Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

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No of samples	Neutron f or energ E (Me	field gy <sup>1)</sup> V)	Reference dose equivalent H (mSv)	Track density N±σ <sub>n-1</sub> (cm <sup>-2</sup> )	Background tracks $N_0 \pm \sigma_{n-1}$ (cm <sup>-2</sup> )	Net track density $N \pm SEM$ (cm <sup>-2</sup> )	Response R±SEM (cm <sup>-2</sup> ·mSv <sup>-1</sup> )
2	0.144	(C)	2.32	3878.9±33.8	380.2 ± 22.5	3498.8 ± 28.7	1508.1 ± 12.3
2	0.250	(C)	1.73	2255.1 ± 40.6	380.2 ± 22.5	1874.9±32.8	1083.8 ± 18.9
2	0.570	(D)	1.50	1222.8 <b>±</b> 28.8	189.6 ± 98.2	$1033.2 \pm 22.1$	688.8 ± 14.7
2	1.200	(B)	2.09	1684.4 ± 178.8	151.6±11.9	1532.8 ± 126.7	733.4 ± 60.7
2	2.850	(E)	2.05	1395.3 ± 228.8	$153.4 \pm 100.6$	1241.9 ± 176.6	605.8 ± 86.2
2	5.300	(D)	2.12	825.8 ± 11.3	189.2 ± 98.2	636.6±69.9	300.3 ± 32.9
2	14.700	(E)	4.20	254.9 ± 2.5	153.4 ± 100.6	801.5±71.1	190.8 ± 16.9
2	Cf-252	(A)	2.27	1189.7 ± 39.4	123.4 ± 1.9	1066.3 ± 27.9	469.7 ± 12.3
2	Cf-252	(B)	2.02	907.2 ± 47.5	151.6 ± 11.9	755.6 ± 34.6	374.1 ± 17.1
2	Cf-252	(C)	2.87	1655.6 ± 86.9	380.2 ± 22.5	$1275.5 \pm 63.5$	444.4 ± 22.1
2	Cf-252	(D)	1.99	1021.2 ± 26.3	189.2 ± 98.2	832.0 ± 71.8	418.1 ± 36.1
2	Cf-252	(E)	2.03	1281.2 ± 38.8	153.4 ± 100.6	1127.8 ± 76.2	555.6 ± 37.6

Table 2: Result of 1986 Joint Irradiation, CR 39 Pershore Moulding

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

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INTERCOMPARISON RESULTS OF THE CENDOS IRRADIATIONS

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#### 1. Description of Dosemeters

dosemeters used for the CENDOS irradiations were The CR-39 track detectors from 3 different batches manufactured by the American Acrylics and Plastics, Inc., in 1982, 83 and 85, respectively. The batches were kept in the open air before the irradiation time in November 1986. The sheets, about 650 µm thick, were covered on both surfaces with polyethylene foils of approximately 45, 50 and 100  $\mu$ m thick for the 1982, 83 and 85 batches, respectively. The sheets, without removing their polyethylene foils, were placed on top of each other with radiator on top of them. Each sheet was devided into 9 pieces of approximately equal sizes. Each irradiation card, 10 x 10 cm, was consisted of 2 or 3 sheets of CR-39 in intimate contact with each other and the radiator.

For fast neutron irradiations, each card was consisted of 3 sheets of CR-39 of different batches and a polyethylene radiator, 2 mm thick.

The card for thermal neutron irradiation was consisted of 2 sheets of CR-39 of the 1985 batch in intimate contact with six boron-loaded scintillators, 2 x 2 cm, with different boron content manufactured by the Bicron Company. The boron concentrations were 1%, 2%, 5% and 10%, respectively. Unfor-

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tunately during the shipment and handling of the dosemeter the boron scintillators have been displaced from the initial CR-39 detector surface, and because of this, the data are not reported here.

#### 2. Processing

The samples were processed by the conventional chemical etching (CE) followed by the electrochemical etching (ECE).

The CE was performed in the 6N KOH solution at 60 °C for 3 hours. At the end of the CE process, the samples were washed in ordinary water for about % h and then in distilled water for about 10 min. The samples were then dried in vacuum to prevent the effect of the dried dropplets on the surfaces which cause spurious counts on the image analyser counting system. The thickness of the chemically etched samples was then measured to an accuracy of  $\pm 1$  µm by am electronic linear gauge manufactured by the Mitutoyo Company.

The ECE process was performed in a thermo-controlled The samples were placed as vessele at 25 °C for 5 hours. separative walls in the electrochemical cells. The two partitions of the cells were filled with 6N KOH and ordniary for the top (irradiated) and bottom surfaces of the water samples, respectively. The positive electrode was connected to The maximum of 10 samples could be the KOH solution. simultaneously. electrochemically etched The applied electrical field was 30 KV/cm with the frequency of 2 KHz. Since the thickness of the samples was different from each other, the samples with approximately equal thicknesses were chosen for each etching run. Therefore, the applied high

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voltage varied from about 1.3 to 2.0 KV RMS depending on the average thicknes of the samples of each run. The elctrochemically etched part of each sample was a circular area with the diameter of about 20 mm. At the end of the ECE process, the samples were washed with distilled water for about 30 min and then dried by tissues.

#### 3. Assessment

The samples were counted by the System III Image Analyser Computer in conjuction with an optical microscope under the magnification of 5 x 10. The counting was performed with green filter to have the tracks with better contrast.

The calibration of the circular area of the field of view was made using a stage micrometer. The experiments showed that the day-to-day calibration was necessary because of the electronic shift of the counting system. It was found that an error of  $\pm 0.01$  mm in the calibration of the field of view results in an error of about  $\pm 4\%$  in the estimation of track density.

A circular area with the diameter of 10 mm cocentric with each sample was scanned and the number of tracks in 289 fields of view were recorded. In order to get the good statistics, the diameter of the sacnning area for background samples was chosen as 12 mm and 416 fields of view were sacnned for each sample.

#### 4. Discussion of results

The results presented in Table I are those of the 1985 batch only. The track density for each neutron energy represents the averaged value of a set of samples. The samples

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with higher track density than the rest of the set were discarded and it was attributed to the scratches on the film.

No inverse square correction was made since it was found to be far less than the statistical errors of the samples. The correction factor for proton contamination during the 14.7 MeV neutron irrdiation at CRNL (W. G. Cross Memorandum, February 19, 1987) does not apply to the present results since the 2 thickness of the polyethylene radiator was about 190 mg/cm.

The average background track density from 5 laboratories 2 was found to be about 100 tracks/cm . This large background was due to the relatively old CR-39 material (1985 batch). The results obtained for the detector responses at different neutron energies are well fitted on the response curve of the CR-39 which has been evaluated previously (Tommasino and Griffith, unpublished data) and shown in Fig. 1.

The responses of the detectors to the Cf-252 irradiations at different laboratories were found to be in good agreement within the standard error of the mean (SEM).

#### Acknowledgements

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Neutron field Reference dose Track density Background Net track Response No of No of or energy 1) equivalent tracks density  $N \pm \sigma_{n-1}$ Background N±SEM  $N_0 \pm \sigma_{n-1}$ R±SEM Samples E (MeV) H (mSv) (cm·2)  $(cm^{-2})$ (cm-2) $(cm-2 \cdot mSv-1)$ Samples (C) 0.144 128.2 + 11.9 65.1 + 5.6 1.97 222.4 ± 21.6 94.2 + 18.5 8 6 0.250(C) 94.2 + 18.5 275.2 + 33.0 152.8 + 18.3 369.4 ± 64.7 8 4 1.801 0.565 (D) 437.5 + 63.3 114.1 + 20.8 323.4 + 33.0 215.9 + 22.0 1.498 5 4 1.200 (B) 577.0 + 62.3 105.5 + 13.3471.5 + 21.4 225.6 + 10.2 7 9 2.09 2.800(E) 104.6 + 19.5 241.8 + 19.9 118.0 + 9.7 346.4 + 35.7 5 2.05 4 5.300 (D) 145.3 + 12.3 68.3 <u>+</u> 5.8 2.117 259.4 + 22.6114.1 + 20.85 8 14.700 (E) 416.4 + 31.1 104.6 + 19.5 311.8 + 16.4 74.2 + 3.9 5 5 4.20 Cf-252 (A) 95.3 + 18.1 396.1 + 15.2 174.5 + 6.7 491.4 + 28.7 5 5 2.27 Cf-252 (B)  $136.0 \pm 12.5$ 274.7 + 25.2 380.2 + 74.2 105.5 + 13.3 7 9 2.02 Cf-252 (C) 361.3 + 32.0 455.5 + 70.0 94.2 + 18.5 163.5 + 14.52.21 8 5 Cf-252 (D) 433.9 + 78.4 114.1 + 20.8 319.8 + 31.1 158.1 + 15.4 5 7 2.023 Cf-252 (E) 442.8 + 75.0 104.6 + 19.5338.2 + 34.7 166.6 + 17.1 5 5 2.03 thermal (A)

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

 $\sigma$  = standard error of the group n-1 SEM = standard error of the mean

\* The incident side of the irradiation card has mistakenly been turned away from the source during the irradiation.

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NRPB Neutron Personal Dosemeter : Results of Joint EURADOS/CENDOS/USA/Canadian Neutron Irradiations 1986

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National Radiological Protection Board, Chilton, Didcot, Oxon, OX11 ORQ, UK

#### 1. DESCRIPTION OF DOSEMETER

Poly allyl diglycol carbonate (PADC) of 2.6% isopropyl peroxydicarbonate (IPP) initiator concentration and 0.2% dioctyl phthalate (DOP) plasticizer was used. Samples were taken from two sheets of a batch stored in dry nitrogen from manufacture by Pershore Mouldings Ltd in April 1986, until preparation of dosemeters by the NRPB Neutron Personal Monitoring Service, as if for routine issue, in October 1986.

Dosemeter packs were irradiated without holders. The detector consists of a flat piece of PADC 27 x 39 mm, of thickness 500  $\mu$ m, uniquely encoded with a BCD number by laser cut holes in an 8 x 4 location matrix. The detector is sealed within an aluminium/ polyethylene laminate pouch. The purpose of sealing detectors inside the pouch is to reduce the response to ambient radon and radon daughter levels, for general protection and, for the present, to enable the use of a pre-existing dosemeter holder.

Only the back surface of the detector is etched. For this surface, the detector itself is the proton radiator for neutron energies up to about 8 MeV. For higher energies, there will be a contribution to the response from the 60  $\mu$ m thick polyethylene lining, the 120  $\mu$ m thick aluminium layer and the surface paper covering of the pouch. A thermal and epithermal neutron response may be obtained via 600 keV initial energy protons produced by capture on nitrogen in a piece of nylon-6 in contact with the rear surface of the detector. However, in this instance, such radiators were not used.

#### 2. PROCESSING

The processing of detectors consists of a chemical pre-etch of one hour at 70°C in 20% w/v NaOH, an electrochemical etch of sixteen hours at 30°C in 20% w/v NaOH with an applied field strength of between 20 and 22 kV cm<sup>-1</sup> r.m.s. of 2 kHz, and a post etch of three hours at 30°C in 20% w/v NaOH.

#### 3. ASSESSMENT

A circular area of approximately 2.5 cm<sup>2</sup> of the back surface of the detector is electrochemically etched. Of this a centralised area of approximate total area of 1.68 cm<sup>2</sup> is counted. This counted area comprises fourteen rectangular sub-areas, 3 x 4 mm. Detectors are read on a fully automated track etch analysis system (TEAS) based on the 40-10 system of Analytical Measuring Systems (AMS) developed by AMS to the specification of NRPB. The read is performed at a magnification of about x40 and allows recognition of pits of diameters greater than about 25 µm. Data from the fourteen sub-areas is used to assess the uniformity, or otherwise, of the pit density distribution. The TEAS reads and decodes the detector identification BCD number.

#### 4. RESULTS

The results for the irradiation are shown in Table 1. The results are also shown in Figures 1 & 2 together with the results for the previous CENDOS irradiations performed in 1985 and for two sets of irradiations made at the National Physical Laboratory. In Figure 1 the results are presented as observed pits per unit area per unit fluence. In Figure 2 the results are converted to response per unit ambient dose equivalent and normalised for each set of irradiations to the response for a californium-252 neutron source.

#### 5. DISCUSSION

The results shown in Figure 1 indicate a significant batch to batch variation in energy dependence of response. This is less marked when the response curves for different batches are normalised to their response to a californium-252 neutron source as illustrated in Figure 2: it is our routine practice to normalise the response of

individual sheets of PADC in this way. The normalisation of the response of individual sheets does not remove within sheet variations in response. Sample to sample variation within a sheet is typically about 10% (one standard deviation) in addition to counting statistics but including variation in response resulting from variability in etch parameters, in particular field strength. The value of the response to californium-252 obtained for the irradiation at CRNL of 56.5  $\pm$  2.9 cm<sup>-2</sup>  $mSv^{-1}$  and at Battelle of 34.3 + 2.5 cm<sup>-2</sup> mSv<sup>-1</sup> are significantly different (double- sided t test at 95% confidence level) from the group mean, 43.3  $\pm$  3.7 cm<sup>-2</sup> mSv<sup>-1</sup>, being 1.3 and 0.8 respectively of the mean value. The reasons for this divergence are not presently understood. There is evidence that the response of PADC is dependent on the degree of polymerisation in such a way that less well polymerised plastic is more radiation sensitive in general and in particular to charged particles of lower energy deposition density (for example higher energy protons) but is more variable in its properties. This hypothesis is being investigated.

#### ACKNOWLEDGEMENTS

The authors wish to thank the members of the NRPB Personal Monitoring Services Group for their assistance in preparation and processing of the dosemeters used in these joint irradiations.



Figure 1 : Dosemeter Response per Unit Fluence

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Figure 2 : Dosemeter Response per Unit Dose Equivalent (ICRP 51 H\*(10))

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No of samples	No of amples E (MeV)		Reference dose equivalent H (mSv)	Track density N±σ <sub>n-1</sub> (cm <sup>-2</sup> )	Background tracks N <sub>0</sub> ±σ <sub>n-1</sub> (cm-2)	Net track density N±SEM (cm-2)	Response R±SEM (cm-2·mSv-1)
4	0.144 (	(C)	2.32	148.5 <u>+</u> 14.3	9.3 <u>+</u> 3.8	139.2 <u>+</u> 7.4	60.0 <u>+</u> 3.2
4	0.250 (	(C)	1.73	204 <u>+</u> 10.4	9.3 <u>+</u> 3.8	195 <u>+</u> 5.5	112.5 <u>+</u> 3.2
4	0.565 (	(D)	1.50	164 <u>+</u> 19.9	6.3 <u>+</u> 1.0	157.7 <u>+</u> 10.0	105.1 <u>+</u> 6.6
4	1.200 (	(B)	2.09	97.5 <u>+</u> 17.5	13.8 <u>+</u> 3.9	83.8 <u>+</u> 8.9	40.1 <u>+</u> 4.3
4	2.800 (	(E)	2.05	89.3 <u>+</u> 11.7	12.3 <u>+</u> 3.3	77.0 <u>+</u> 6.1	37.6 <u>+</u> 3.0 ·
4	5.300 (	(D)	2.12	56 <u>+</u> 9.1	6.3 <u>+</u> 1.0	49.7 <u>+</u> 4.1	23.4 <u>+</u> 1.9
4	14.700 (	(E)	4.20	207.3 <u>+</u> 8.2	12.3 <u>+</u> 3.3	195 <u>+</u> 4.4	46.5 <u>+</u> 1.0
4	Cf-252 (	(A)	2.27	124.3 <u>+</u> 49.5	23.5 <u>+</u> 13.0	100.8 <u>+</u> 25.6	44.4 <u>+</u> 11.3
4	Cf-252 (	(B)	2.02	83.0 <u>+</u> 9.4	13.8 <u>+</u> 3.9	69.3 <u>+</u> 5.1	34.3 <u>+</u> 2.5
4	Cf-252 (	(C)	2.87	122.3 <u>+</u> 16.4	9.3 <u>+</u> 3.8	113 <u>+</u> 8.4	39.4 <u>+</u> 2.9
4	Cf-252	(D)	1.99	90.3 <u>+</u> 18.6	6.3 <u>+</u> 1.0	84.0 <u>+</u> 8.7	42.1 <u>+</u> 4.4
4	Cf-252 (	(E)	2.03	127 <u>+</u> 11.2	12.3 <u>+</u> 3.3	114.7 <u>+</u> 5.8	56.5 <u>+</u> 2.9
	thermal	(A)					

## Table 1 : Results of 1986 Joint Irradiation

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

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SUMMARY OF THE RESULTS OBTAINED BY ENEA-BOLOGNA IN JOINT EUROPEAN - U.S. - CANADIAN NEUTRON DOSIMETER IRRADIATIONS.

## O. Civolani and L. Lembo Laboratorio Applicazioni di Dosimetria ENEA Bologna (Italy)

1- Description of dosimeters.

Two different types of neutron dosimeters both based on CR-39 detectors were used during these irradiations. The first, with CR-39 detectors chemically etched after the irradiations, was the same type used in the previous Cendos irradiations, that is, a flat CR-39 plastic 30 mm by 12 mm and 1.5 mm thick, covered on both sides with a polyethylene converter 1 mm thick. The CR-39 polymer was produced in our laboratory using a high purity PPG monomer with 6% CHPC,0.2 % DOP and a curing time of 96 h at 70 °C.

The second was a prototype of the dosimeter based on a chemical and electrochemical etching recently to developed for our routine appli- cations. The reason for this choice is mainly related to the persisting difficulty in finding a high quality commercial CR-39 polymer having a sufficiently low and reproducible background with the chemical etching process. The detector consists of a CR-39 card 25 mm by 30 mm and 600 um thick. No additional radiator was used because only the back surface of the detector was electrochemically etched. The CR-39 plastic used in this experience was taken from a batch manufactured by American Acrylics in the first month of 1986, with adhesive polyethylene protective sheet on both sides. 2- Processing.

The CR-39 detectors employed in the first type of dosimeter were processed using a traditional chemical etching with a sodium hydroxide solution 6.25 N at 70 °C and an etching time of 8 hours.

For the second type of dosimeter the detectors were placed in a lucite cell containing a potassium hydroxide solution 6N and etched inside an oven with a fixed temperature of 60 °C, in the following conditions, suggested by Dale Hankins :

A)	3000	Volts	-	60	Ηz	for	6 ł	nours	
B)	3000	Volts	-	2000	Hz	for	20	min.	
C)	-			-		for	15	min.	

The three etching steps were performed one immediately after the other using an HP41CX pocket computer connected to a Homann-Bell high voltage supply, by which it is possible to programme the complete etching cycle.

Only the rear surface of the detectors was etched.

3. Assessment.

The chemical etched samples were evaluated using a Zeiss optical microscope with a magnification of 250. For each sample the tracks over a total surface of 1.5 mm<sup>2</sup> were counted.

Track evaluation of the electrochemical etched samples was done by means of an inexpensive image analysis system, AMS 40-10, connected through a videcon tube with a low-power microscope. Total magnification on the televisin monitor was about 70. For each sample a total surface of about 1 cm<sup>2</sup> was observed.

4. Results

Table 1 and 2 report the results obtained during this neutron irradiation exercise with CR-39 dosimeters processed with chemical and electrochemical techniques respectively. No data are reported for thermal neutrons, since in this case thermoluminescence detectors are used in our personnel dosimetry service.

#### 5 - Comments.

The results obtained during this experience with chemically etched detectors have substantially confirmed the sensitivity and energy response obtained with the same technique during the previous Cendos experience, despite the fact that the CR-39 polymers used in the two experiences were produced by different manufacturers. An exception was the significant sensitivity reduction of about 30 % obtained this time at the energy of 14.7 MeV.

As for the electrochemical etched detectors, the results have confirmed that the response in the low energy region is significantly better than that of the normal etching technique. Here too some investigation must be done to improve the 14.7 MeV energy response. In addition these results show a CR-39 sensitivity significantly higher than that obtained by other laboratories that used electrochemical procedures during the previous Cendos experiment, but comparable with that obtained by Tommasino who used the same technique suggested by Hankins.

#### TABLE 1 - RESULTS OF CHEMICALLY ETCHED DETECTORS

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N° of samples	Neutron energy (1) : ( MeV)	Reference dose equivalent H (mSv)	Track density N + σ <sub>n-1</sub> ( cm <sup>-2</sup> )	Background tracks (2) N <sub>o</sub> + O <sub>n-1</sub> ( cm <sup>-2</sup> )	Net track density N <sub>N</sub> + SEM ( cm <sup>-2</sup> )	Response density R + SEM ( cm <sup>-2.</sup> mSv <sup>-1</sup> )
			- · ·			
4	0.142 (C)	1.997	1500 ± 390	650 ± 188	850 ± 216	426 ± 108
4	0.248 (C)	1.773	2060 ± 597	650 ± 188	1410 ± 312	795 ± 180
4	0.570 (D)	1.499	2466 ± 197	650 ± 188	1816 ± 136	1211 ± 91
4	1.200 (B)	2.09	2966 ± 326	650 ± 188	2316 ± 188	1108 ± 89
4	2.850 (E)	2.05	2566 ± 257	650 ± 188	1916 ± 159	934 ± 77
4	5.300 (D)	2.117	2333 ± 466	650 ± 188	1683 ± 251	795 ± 118
4	14.71 (E)	4.20	2340 ± 450	650 ± 188	1690 ± 244	402 ± 58
4	Cf-252 (A)	2.27	2560 ± 385	650 ± 188	1910 ± 214	841 ± 94
4	Cf-252 (B)	2.02	2950 ± 300	650 ± 188	2300 ± 177	1139 ± 88
4	Cf-252 (C)	2.21	2800 ± 728	650 ± 188	2150 ± 376	973 ± 170
4	Cf-252(D)	1.987	2600 ± 520	650 ± 188	1950 ± 276	981 ± 139
4	Cf-252 (E) Thermal (A)	2.03	2500 <u>+</u> 450 	650 ± 188	1850 ± 244 	911 ± 120 

1) - IRRADIATION AT HARWELL (A), BATTELLE(B), PTB(C), GSF(D), CRNL(E) 2) NUMBER OF BACKGROUND SAMPLES : 4

N° of samples	Neutron energy (1)	Reference dose	Track density	Background tracks (2)	Net track density	Response density
	(MeV)	equivalent H (mSv)	N + o <sup>-1</sup> ( cm <sup>-2</sup> )	N <sub>o</sub> + 6 <sup>°</sup> n-1 ( cm <sup>-2</sup> )	N + SEM ( cm <sup>-2</sup> )	R + SEM ( cm <sup>-2,</sup> mSv <sup>-1</sup> )
4	0.142 (C)	1.997	3237 ± 129	1290 ± 193	1947 ± 116	974 <u>±</u> 58
4	0.248 (C)	1.773	2900 ± 174	1290 ± 193	1610 ± 130	908 ± 73
4	0.570 (D)	1.499	2383 ± 119	1350 ± 121	1033 ± 85	689 ± 57
4	1.200 (B)	2.09	2175 ± 130	1312 ± 196	863 ±117	413 ± 56
4	2.850 (E)	2.05	2185 ± 284	1122 ± 168	1063 ±165	518 ± 80
4	5.300 (D)	2.117	2275 ± 205	1350 ± 121	925 ±119	437 ± 56
4	14.71 (E)	4.20	1925 ± 173	1122 ± 168	803 ±120	191 ± 28
4	Cf-252 (A)	2.27	2350 ± 223	1382 ± 180	968 ±143	426 ± 63
4	Cf-252 (B)	2.02	2222 ± 178	1312 ± 196	910 ±132	450 ± 65
4	Cf-252 (C)	2.21	2450 ± 465	1290 ± 193	1160 ±251	525 ± 113
4	Cf-252(D)	1.987	1917 ± 77	1350 ± 121	567 ±72	285 ± 36
4	Cf-252 (E) Thermal (A)	2.03	2025 ± 405 	1122 ± 168	903 ±219	445 ± 108 

## TABLE 2 - RESULTS OF ELECTROCHEMICALLY ETCHED DETECTORS

1) - IRRADIATION AT HARWELL (A), BATTELLE(B), PTB(C), GSF(D), CRNL(E) 2) 4 BACKGROUND SAMPLES FOR EACH IRRAD. CENTER

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## RESPONSE OF CRNL NEUTRON DOSEMETERS IN JOINT EUROPEAN/US/CANADIAN IRRADIATIONS

W.G. Cross, A. Arneja and J.L. Kim

#### 1. Description of Dosemeters

The detectors were cut from a sheet of "dosimeter grade" (93% monomer purity) CR-39 manufactured by American Acrylics in July, 1986. Each sheet is covered by a protective layer of polyethylene, about 12  $mg/cm^2$  thick. Prior to irradiation the CR-39 was stored in air, in darkness and at room temperature. Detectors were 0.63 mm thick and approximately 3 cm x 3 cm. Four detectors were exposed at each energy, stacked in pairs and sealed in an envelope of polyethylene, 12  $mg/cm^2$  thick. Four "background" dosemeters accompanied each set of irradiated dosemeters.

The backgrounds on the two sides of the detectors differed by a factor of about 11. The low-background side  $(30 \pm 12 \text{ tracks/cm}^2)$  was towards the neutrons and was used for fast neutron measurements. For irradiations with 5.3 and 14.7 MeV neutrons, additional polyethylene radiators were in front of each detector, the total thickness of polyethylene being 220 mg/cm<sup>2</sup> for 14.7 MeV, 48 mg/cm<sup>2</sup> at 5.3 MeV and 24 mg/cm<sup>2</sup> lower energies. Part of the high-background (rear) side of each detector was against a thermal neutron converter (Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> in teflon) and was used to measure the thermal neutron fluence.

## 2. Processing

Detectors were pre-etched for 1.5 hours, exposed overnight to water vapour at  $60^{\circ}$ C, electrochemically etched for 5 hours with a 60 Hz field of 20 kV (rms)/cm and for 45 minutes with a 2 kHz field of the same strength. All etching was in 6N KOH at  $60^{\circ}$ C. Both CR-39 surfaces were etched.

#### 3. Assessment

Tracks were magnified 76 times in a microfiche reader (3M model 297-BH) and counted by eye. About 0.4 cm<sup>2</sup> area was counted. The minimum diameter of counted tracks was about 0.5 mm.

#### 4. Discussion of Results

The results for polyethylene radiators are given in Table 1. The errors given are only those from counting statistics. The standard error of the background corresponds to about 20 microsieverts (2 mrem). For the measurements at 5.3 MeV, the dosemeters were mounted on the card with (by mistake) the high-background side of the CR-39 towards the neutrons. The background is therefore much larger and the measured response is less accurate than at other energies.

These results are compared in Figure 1 with a calculated response, obtained under the assumptions that only recoil protons between 0.08 and 2.75 MeV produce tracks. These limits are somewhat narrower than those for which we have previously calculated the response (1) because for the present measurements the field strength used for electrochemical etching was lower and pre-etching was used, both in order to reduce background. An overall error of  $\pm 15\%$  is assumed for the measurements. The agreement between measured and calculated values is reasonable except at 14.7 MeV, where a substantial fraction of the response is known to come from alpha particles and heavy recoils, and at 1.2 MeV, where we do not know the cause of the discrepancy.

We also do not know the cause of the unexpectedly high variations in the response to  $^{252}\mathrm{Cf}$  neutrons.

The response to thermal neutrons agrees to  $\pm 3\%$  with the value that we would normally use, for the same etching conditions, in calculating thermal neutron dose equivalents. While such high sensitivity is sometimes useful in detecting weak fields, it is far from ideal for measuring significant dose equivalents and we are looking for suitable converter material with a lower boron content.

Radiators of Au and of Al were put against parts of the areas of the fast neutron detectors, with the objective of determining the response of the etched layer alone, at each energy. This experiment was unsuccessful, because the background arising from alpha-emitting contaminants in (or on the surface of) these radiators was too high to obtain significant values of the etched layer response. To do this experiment it would be necessary to have the metal radiators against the CR-39 for much shorter times.  (1) Cross, W.G., Arneja, A. and Ing, H. The response of electrochemically-etched CR-39 to protons of 10 keV to 3 MeV, Nuclear Tracks, <u>12</u>, 649-652 (1986).



Fig. 1. Measured and calculated response of CR-39. The curve is calculated on the assumptions that only recoil protons of energies between 0.08 and 2.75 MeV produce visible tracks.

No of	Neutron field or energy <sup>1)</sup>	Reference dose equivalent	Track density	Background tracks	Net track density	Response
samples	E (MeV)	H (mSv)	$\frac{N \pm \sigma_{n-1}}{(cm^{-2})}$	$\frac{N_0 \pm \sigma_{n-1}}{(cm^{-2})}$	$\frac{N \pm SEM}{(cm^{-2})}$	R±SEM (cm-2·mSv-1)
4	0.144 (C)	2.287	1482 <u>+</u> 61	30 <u>+</u> 12	1452 <u>+</u> 62	635 + 27
4	0.249 (C)	1.715	1162 <u>+</u> 54	30 <u>+</u> 12	1132 <u>+</u> 55	660 <u>+</u> 31
4	0.565 (D)	1.498	1148 <u>+</u> 54	30 + 12	1118 <u>+</u> 55	746 <u>+</u> 36
4	1.200 (B)	2.07	1445 <u>+</u> 60	30 <u>+</u> 12	1415 <u>+</u> 61	683 + 29
4	2.800 (E)	2.03	2207 <u>+</u> 74	30 <u>+</u> 12	2177 <u>+</u> 75	$1072 \pm 36$
4	5.300 (D)	2.12	1287 + 56	330 <u>+</u> 47	957 <u>+</u> 73	451 + 62
4	14.700 (E)	4.20	1836 <u>+</u> 68	30 <u>+</u> 12	1806 <u>+</u> 69	$430 \pm 16$
	•					
4	Cf-252 (A)	2.11	1430 <u>+</u> 60	30 + 12	1400 + 61	664 + 27
4	Cf-252 (B)	2.01	1273 <u>+</u> 56	30 + 12	1243 <u>+</u> 5,7	618 <u>+</u> 27
4	Cf-252 (C)	2.82	1360 <u>+</u> 59	30 <u>+</u> 12	1330 <u>+</u> 60	472 + 21
4	Cf-252 (D)	1.98	1026 <u>+</u> 50	30 + 12	996 <u>+</u> 51	503 <u>+</u> 26
4	Cf-252 (E)	2.04	1480 <u>+</u> 61	30 <u>+</u> 12	1450 <u>+</u> 62	714 <u>+</u> 30
1	thermal (A)	4.73	148560 <u>+</u> 1411	30 <u>+</u> 12	148530 <u>+</u> 1411	31402 + 299

<sup>1)</sup> Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

# LEPOFI -Experimental results of CENDOS neutron irradiations.

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## <u>1 - Description of dosemeter :</u>

The differential structure dosemeter (Fig.1) has been tested during last joint neutron irradiations of CENDOS for fast (0.12–14.7 MeV, Cf) and thermal neutrons.

The differential method consists in irradiating two areas of the detector, one of these covered by a radiator, the other one without a radiator, gives the response for the detector alone, then in substracting the two responses.

## 2 - Experimental conditions :

Two kinds of detector are used :

- CR 39 (Pershore Moulding Corporation)

- C.A.D.(Société ESSILOR - France) which is a

similar product that we calibrated. Before using, C.A.D. is pre-etched (1 hour in mixture of 60% (volume)ethanol, 40% 6.25N NaOH at 70°C).

Our track etching conditions are the following :

- NaOH, 6.25 N, 60° C

- the etching time is optimized according to the results of a previous study.

Our measures are carried out using an optical microscope (x 250, x 400) with statistical counting.



Figure n° 1: Dosemeter used for -a) fast neutron detection with or without polyethylene (35µm) radiator -b) thermal neutron detection

<u> 3 - Results</u> :

Our experimental results for the normal incidence are presented in Tab.1 for CR 39 and in Tab.2 for C.A.D.

We plot, on figure 2, the experimental values of response, for neutron energies : 0.144, 0.250, 0.570, 1.2, 2.85 MeV and for irradiations with californium. The discrepancies between experimental and calculated values of effective efficiency\* are greater for small energies, and can be reduced when the etching time is ajusted. In this case, we can see that the response of CR 39 and C.A.D. are similar.

(\*)- The effective efficiency corresponds to the calculated number of protons from the radiator which are registered in the detector.





d - 1.2 MeV e - 2.85 MeV f - Californium 252

## <u> 4 - Remark :</u>

We made a note of important discrepancies between the background in different laboratories.

<u>5 - Aknowledgements</u>

We are grateful HARWELL, PTB, GSF, CNRL, BATTELLE-laboratories to this organization of neutron irradiations.

No of samples	Neutron field or energy <sup>1)</sup> E (MeV)	Reference dose equivalent H (mSv)	$\begin{array}{c} {\rm Track\ density}\\ {\rm with\ radiator}\\ {\rm N\ \pm\ \sigma_{n-1}}\\ {\rm (cm^{-2})} \end{array}$	Track density without radiator $N_0 \pm \sigma_{n-1}$ (cm-2)	Net track density N±SEM (cm-2)	Response R±SEM (cm <sup>-2</sup> ·mSv <sup>-1</sup> )
3	0.144 (C)	2.066	6838 <u>+</u> 265	6000 + 240	838 <u>+</u> 206	406 <u>+</u> 100
3	0.250 (C)	1.732	8339 <u>+</u> 346	7875 + 306	464 <u>+</u> 267	268 <u>+</u> 154
3	0.565 (D)	1.498	4719 <u>+</u> 222	- 3630 <u>+</u> 288	1089 <u>+</u> 210	727 <u>+</u> 140
3	1.200 (B)	2.09	5200 <u>+</u> 235	3427 <u>+</u> 268	1773 <u>+</u> 206	848 <u>+</u> 99
3	2.800 (E)	2.05	4867 <u>+</u> 221	3721 <u>+</u> 240	1146 <u>+</u> 188	559 <u>+</u> 92
3	5.300 (D)	2.12	2840 <u>+</u> 280	2120 <u>+</u> 235	720 <u>+</u> 211	340 <u>+</u> 100
3	14.700 (E)	4.2	3989 <u>+</u> 401	-	-	-
3	Cf-252 (A)	2.27	4125 <u>+</u> 148	2681 <u>+</u> 151	1444 <u>+</u> 122	636 <u>+</u> 54
	Cf-252 (B)					_
	Cf-252 (C)					
	Cf-252 (D)					
	Cf-252 (E)					
			radiator + <sup>10</sup> B	radiator		
3	thermal (A)	2.64	4400 <u>+</u> 222	1813 <u>+</u> 140	2587 <u>+</u> 152	980 <u>+</u> 57

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

Background tracks: (A) -
$$1200 \pm 385$$
(D)- $2040 \pm 310$ (B) - $1220 \pm 240$ (E)- $1320 \pm 300$ (C) - $2280 \pm 280$ Limoges - $1200 \pm 350$ 

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No of samples	Neutron field or energy <sup>1)</sup> E (MeV)	Reference dose equivalent H (mSv)	$\begin{array}{c} {\rm Track\ density}\\ {\rm with\ radiator}\\ {\rm N}\pm\sigma_{n-1}\\ ({\rm cm}^{-2}) \end{array}$	Track density without radiator $N_0 \pm \sigma_{n-1}$ (cm-2)	Net track density $N \pm SEM$ (cm <sup>-2</sup> )	Response R±SEM (cm <sup>-2</sup> ·mSv <sup>-1</sup> )
3	0.144 (C)	2.066	8882 <u>+</u> 335	7559 <u>+</u> 215	1323 <u>+</u> 230	640 <u>+</u> 111
3	0.250 (C)	1.732	7880 <u>+</u> 346	7441 <u>+</u> 263	439 <u>+</u> 251	254 <u>+</u> 145
3	0.565 (D)	1.498	4573 <u>+</u> 244	3681 <u>+</u> 240	892 <u>+</u> 198	596 <u>+</u> 132
3	1.200 (B)	2.09	5413 <u>+</u> 200	3739 <u>+</u> 306	1674 <u>+</u> 211	801 <u>+</u> 101
3	2.800 (E)	2.05	5139 <u>+</u> 216	4079 <u>+</u> 164	1060 <u>+</u> 157	517 <u>+</u> 76
3	5.300 (D)	2.12	3840 <u>+</u> 260	3081 <u>+</u> 320	759 <u>+</u> 238	358 <u>+</u> 113
3	14.700 (E)	4.2	4308 <u>+</u> 346	-	-	-
3	Cf-252 (A) Cf-252 (B) Cf-252 (C) Cf-252 (D) Cf-252 (E)	2.27	3641 <u>+</u> 127 radiator + <sup>10</sup> B	2440 <u>+</u> 145 radiator	1201 <u>+</u> 111	529 <u>+</u> 49
3	thermal (A)	2.64	4867 <u>+</u> 225	1613 <u>+</u> 193	3254 <u>+</u> 172	1233 <u>+</u> 65

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

Background tracks : (A) -	1260	<u>+</u> 310	(D)-	2160	<u>+</u> 360
(B)-	1140	<u>+</u> 240	(E)-	1440	<u>+</u> 280
(C) -	2380	<u>+</u> 310	Limoges -	1250	<u>+</u> 350

#### SUMMARY OF RESULTS OBTAINED AT HARWELL USING ELECTROCHEMICAL ETCHING OF COMMERCIAL GRADE CR-39

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#### Description of Dosemeters

One sheet of CR-39 material was used, taken from a batch manufactured in April 1986 by Pershore Mouldings Ltd (Standard Grade, 2.6% IPP, 0.2% DOP, 32 hour cure cycle, 500  $\mu$ m thick). The sheet was supplied covered on both sides with a polyethylene protective film approximately 50  $\mu$ m thick. One side of the CR-39 sheet was identified by marking the covering film with an indelible pen, prior to laser-cutting the sheets into 22 mm square samples. The dosemeter samples were then stored in a dry nitrogen atmosphere before use. Four samples were stacked in pairs in polyethylene/ /aluminium/paper pouches, which were heat-sealed in ambient air. Two pouches were mounted side by side (on a single card) for each irradiation. No additional radiators were used, although only the back surface of the plastic was electrochemically-etched, so that the thinnest radiator effectively consisted of the paper/aluminium/ polyethylene pouch in front of  $\simeq 500 \ \mu m$  of CR-39.

Eight background samples accompanied each batch of samples to each irradiation laboratory. These were processed with the exposed samples.

#### Processing

Samples were removed from the sealed pouches immediately prior to processing, in batches of up to 40. The processing conditions were as follows:-

pre-etch 6.25N KOH, 35<sup>O</sup>C 3 hours

ECE 6.25N KOH,  $35^{\circ}$ C 9 hours, 2kHz, 21kV (rms) cm<sup>-1</sup> post-etch 6.25N KOH,  $35^{\circ}$ C 10½ hours

(only the rear surface of each sample was electrochemically-etched). Recent work has shown that this process leads to a better low-energy and angular response than the process used previously.

#### Assessment

The samples were counted using a Kodak Carousel S-AV 2010 slide projector set up for approximately X30 magnification. Spots having the characteristic circular shape (with starred edges), and with diameters greater than ~10  $\mu$ m were counted manually. An area of 1 cm<sup>2</sup> was counted for each sample, using a perspex screen covered with a sheet of translucent drawing film divided into known areas to make counting easier. The image was projected onto the screen and counted from behind it.

#### Results

The results are summarised in Table 1. Cards 1-4 were sent to CRNL, cards 5-8 to PTB, 9-11 to Battelle, 12-15 to GSF and 16-18 to Harwell; cards 4,8,11,15 and 18 were used as background controls. All the spot distributions were uniform to the eye and the statistics were normal but with standard deviations generally up to twice those expected from Poisson statistics. No attempt was made to correct for effects resulting from variability in the thickness of the plastic sheet, with consequent variations in the electric field applied to each sample (<5%).

## Discussion

No differences were detected between the pairs of samples at the front and back of the sealed pouches over the whole range of neutron energies.

It can be noticed that the European sets of backgrounds are fairly low whereas the backgrounds on samples which crossed the Atlantic by air are slightly higher. This is probably due to the effect of cosmic ray neutrons during the outward and return flights.

The Harwell response to <sup>252</sup>Cf can be seen to be low by about 25% compared to the others. The reason for this is not understood, as the samples were processed together. It is thought that the loss of sensitivity is due to some uncontrolled environmental effect which 'aged' the plastic in some way.

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CARD	No of	Neutron field or energy <sup>1)</sup>	Reference dose equivalent	Track density	Background tracks	Net track density	Response R+SEM
No.	samples	E (MeV)	H (mSv)	$(\text{cm}^{-2})$	$(cm^{-2})$	$(cm^{-2})$	$(cm^{-2} \cdot mSv^{-1})$
5	8	0.144 (C)	1.97	251 <u>±</u> 33	14 <u>+</u> 3	237 <u>+</u> 12	120 <u>+</u> 6
6	8	0.250 (C)	1.80	343 ± 40	14 <u>+</u> 3	329 <u>+</u> 14	183 <u>+</u> 8
12	8	0.565 (D)	1.50	296 <u>+</u> 34	19 <u>+</u> 4	277 <u>+</u> 12	185 ± 8
9	8	1.200 (B)	2.09	288 ± 48	24 ± 7	264 <u>+</u> 17	126 ± 8
1	8	2.800 (E)	2.05	194 ± 13	24 <u>+</u> 7	170 <u>+</u> 4	83 <u>+</u> 2
13	8	5.300 (D)	2.12	138 <u>+</u> 22	19 <u>+</u> 4	119 <u>+</u> 8	56 <u>+</u> 4
2	8	14.700 (E)	4.20	249 <u>+</u> 23	24 <u>+</u> 7	225 <u>+</u> 8	54 ± 2
16	8	Cf-252 (A)	2.27	209 <u>+</u> 17	19 ± 4	190 <sub>±</sub> 6	84 <u>+</u> 3
10	8	Cf-252 (B)	2.02	251 <u>+</u> 28	24 <u>+</u> 7	227 <u>+</u> 10	112 ± 5
7	8	Cf-252 (C)	2.21	254 <u>+</u> 22	14 ± 3	240 <u>+</u> 8	109 ± 4
14	8	Cf-252 (D)	1.99	247 ± 17	19 ± 4	228 <u>+</u> 7	115 ± 4
3	8	Cf-252 (E)	2.03	258 ± 11	24 <u>+</u> 7	234 <u>+</u> 5	115 <u>+</u> 2
17	8	thermal (A)	2.64	32 🛨 5	19 <u>+</u> 4	13 <u>+</u> 2	*

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

NB:  $\sigma n-1$  = standard deviation of group

SEM = standard error of mean of group

\* Response  $\Im$  nil after subtraction of accompanying fast dose contribution

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Results obtained at LLNL from the European/US/Canadian Neutron Dosimeter Irradiations-Nov 1986

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#### 1. Description of Dosimeter.

The CR-39 foils used in this study were from a sheet made by American Acrylics in September, 1986. The CR-39 foils were placed in personnel neutron badges of the type that is routinely worn by personnel at the Lawrence Livermore National Laboratory (LLNL). The CR-39 foils are about 0.025 in. thick and had a 0.005 in. thick polyethylene protective covering on both sides of the foils. The foils are stored in the dark inside a refrigerator prior to being issued. Three foils are stacked and placed into a recess in the back of the badge. The side of the foil to be etched is oriented toward the person. This orientation is used to improve the directional dependence of the dosimeter. A security badge, beta shield and Panasonic TLD holder are located between the foils and the source. No light can reach the foils when they are in the badge. Two badges (6 foils) were sent for each exposure but in most cases only 5 of the foils were etched and the results used in this study. The others are being held for track-size distribution studies.

2. Processing

We used our standard electrochemical etching procedure shown in Table 1 to process the foils.

#### 3. Assessment.

The foils were read using a Biotran Colony counter. Six fields of view,  $3 \times 3 \text{ mm}$  in size, were read traversing down the center of the CR-39 foils. The average of these 6 readings was used to determine the track density in tracks/cm<sup>2</sup> on the CR-39 foil.

#### 4. Discussion of results.

The results obtained in this study are given in Table 2 and have been plotted in Fig. 1. The dashed line shown in Fig. 1 is based on a recently completed study of the energy dependence. The position of this curve was normalized to the average of the data points obtained in this study. There is good agreement between the data and the curve except for the points at 144 keV and at 2.85 MeV. The point at 144 keV is about 25% low but is of little concern because it is near the low energy cut-off and several factors, such as a small difference in foil thickness, could have caused the low reading. The point at 2.85 MeV is 20% low which is much lower than we would expect to find at this energy. At this time the reason for the low reading is not known.

The results obtained with the <sup>252</sup>Cf sources are given in Fig. 1 and show good agreement except for the foils exposed at Battelle where the results are about

20% higher than the average obtained with the other <sup>252</sup>Cf sources. The reason for the difference is not known but may have been experimental error during the exposure. We always include in each etch chamber several control foils that have been exposed to our <sup>252</sup>Cf source. This eliminates variations in the etching process as a possible explanation for the observed difference.

Our CR-39 foils have no response to thermal neutrons. The CR-39 foils exposed in the thermal neutron beam had a track density equivalent to 0.09 mSv from the fast neutrons in the beam. This compares to the reported fast neutron dose in the beam of 0.26 mSv. It may be that the lower energy threshold (about 150 keV), is too high to detect all of the fast neutron dose.

The Biotran Colony counter was also used to determined the track-size distribution on the foils as a part of another study of track-size distributions vs neutron energy. The results are given in Fig. 2 and confirm the reported energies of the incident neutrons are correct and that the badges were not interchanged during calibration. This track size study is investigating the effect various etching parameters have on the track-size distributions. Our standard etching procedure, which was used on these foils, does not give as good a track-size resolution as that obtained using other etching procedures.

	Etching	Blow up	Post Etch
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

Table 1. Etching parameters used in this intercomparison.

\* 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 between 0.022 and 0.029 inches.



Fig. 1 The results obtained in this study are shown as open circles and the dashed line is the energy dependence curve. The result with the various Cf sources are also shown.



Fig. 2 The track-size distribution obtained from the CR-39 foils exposed in this study.

Badge Numbers	No of samples	Neutron field or energy <sup>1)</sup>	Reference dose equivalent	Track density N±σ <sub>n-1</sub>	Background tracks No±go-1	Net track density N±SEM	Response $R\pm SEM$
	-	E (MeV)	H (mSv)	$(\operatorname{cm}^{-2})$	(cm-2)	(cm-2)	$(cm^{-2} \cdot mSv^{-1})$
31-32	5	0.144 (C)	1.97	671 ± 197	58 ± 10	613 ± 88	311 ± 45
33-34	5	0.250 (C)	1.801	1009 ± 44	58 ± 10	951 ± 21	528 ± 19
15-16	6	0.565 (D)	1.498	803 ± 39	54 ± 14	749 ± 17	500 ± 11
23-24	5	1.200 (B)	2.09	1089 ± 113	39 ± 12	1050 ± 51	502 ± 24
9-10	5	2.800 (E)	2.05	854 ± 103	48 ± 25	806 ± 47	393 ± 23
17-18	6	5.300 (D)	2.120	731 ± 31	54 ± 14	678 ± 14	320 ± 6.6
7-8	5	14.700 (E)	4.20	991 ± 69	48 ± 25	943 ± 32	224 ± 7.6
5-6	4	Cf-252 (A)	2.27	1004 ± 91	49 ± 16	955 ± 30	421 ± 13
25-26	5	Cf-252 (B)	2.02	1030 ± 67	39 ± 12	991 ± 30	491 ± 15
29-30	4	Cf-252 (C)	2.21	973 ± 41	58 ± 10	915 ± 16	414 ± 7.4
19-20	6	Cf-252 (D)	1.994	827 ± 83	54 ± 14	773 ± 34	388 ± 17
13-14	5	Cf-252 (E)	2.03	873 ± 58	48 ± 25	825 ± 28	406 ± 14
1-2	5	thermal (A)	2.63	84 ± 32	49 ± 16	84 ± 27	32 ± 10

Table 2. Results obtained from CR-39 foils.

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

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EURADOS-CENDOS joint neutron irradiation, 1986-1987. Results from Berkeley Nuclear Laboratories.

J. R. Harvey and A. R. Weeks. CEGB, Berkeley Nuclear Laboratories, Berkeley, Gloucestershire GL13 9PB. England.

#### DESCRIPTION OF THE DOSEMETER

The CR39 was purchased from Tastrak Bristol. Each piece was 20mm by 10mm and 1.8-2.0 mm thick, and carried an engraved code number. The results quoted here were for samples irradiated in moulded polypropylene holders. The holders have overall dimensions of approximately 30mm by 30mm and hold two pieces of CR39. Wall thickness is in the range 2.0mm. to 2.7mm. One piece of CR39 was in close contact with a piece of BN1, a material marketed by Kodak which consists of a layer of borated material of thickness about 80 microns on a polyester base. This ensures that one element has high sensitivity to thermal and near-thermal neutrons.

#### PROCESSING

After exposure to neutrons each detector (except those used for thermal neutron detection) was given a "pre-etch" of one hour in a mixture of 60% (volume) methanol, 40% 6.25 N sodium hydroxide at 70°C. This treatment removes approximately 60 microns from each face and polishes the surface. The detectors were then immediately immersed in 6.25 N sodium hydroxide at 70°C for six hours, washed, dried and read out.

#### ASSESSMENT

Light from a quartz-halogen projector bulb is channelled, via a light guide, into one edge of the CR39 detector. The light is totally internally reflected from the faces of the CR39 except where a defect or pit exists where it is reflected and refracted. Pits and defects therefore appear as bright spots of light when the CR39 is viewed from a position normal to its surface. The surface is viewed by a videcon tube through a low-power microscope. The signal from the videcon tube is fed to a television monitor and inexpensive image analysis system which incorporates an auto-focus system. The intensity of the light is so great that the videcon tube registers images much larger than the true pit size. The total number of pits within an area in

the range 1  $cm^2$  to 1/128  $cm^2$  are counted typically in 10 seconds. The system is described in detail elsewhere<sup>(1)</sup>

We have recently found that the low energy threshold can be reduced if the sample is viewed at an angle to the normal rather than normal to the surface, and measurements at 20<sup>°</sup> viewing angle are reported here.

#### The Readings

CR39 samples exposed to fast neutrons were read on both sides and an average value taken; those exposed to thermal neutrons were read on the side adjacent to the boron radiator. The results are summarized in the two tables.

#### DISCUSSION

The results are very similar to those found in previous experiments<sup>(1)</sup>, although it is clear that the low energy threshold can be reduced by non-normal viewing. This reduction of the low energy threshold is associated with an increase in background when expressed in dose equivalent terms. It can be seen that the sensitivity to thermal neutrons is very high when the BN1 radiator is used. Data from experiments made with foils prepared from diluted boron, which are more appropriate for radiological protection applications, are being prepared for publication.

#### REFERENCES

1. Harvey J. R. and Weeks, A. R. Progress towards the Development of a Personal Neutron Dosimetry System Based on the Chemical Etch of CR39. U.K. C.E.G.B. Report TPRD/B/0851/R86. (1986)

Viewing angle 0°

No of samples	Neutron field or energy1) E (MeV)	Reference dose equivalent H (mSv)	Track density N±σ <sub>n-1</sub> (cm-2)	Background tracks N <sub>0</sub> ±σ <sub>n-1</sub> (cm <sup>-2</sup> )	Net track density N±SEM (cm-2)	Response R±SEM (cm- <sup>2</sup> ·mSv-1)
4	0.144(C)	2.0	0			
4	0.25(C)	1.73	520 <u>+</u> 11	49 <u>+</u> 12	471 <u>+</u> 6	272 <u>+</u> 3
4	0.57(D)	1.5	501 <u>+</u> 44	49 <u>+</u> 12	452 <u>+</u> 22	301 <u>+</u> 15
4	1.20(B)	2.09	738 <u>+</u> 22	49 <u>+</u> 12	689 <u>+</u> 11	330 <u>+</u> 5
4	2.85(E)	2.05	664 <u>+</u> 61	49 <u>+</u> 12	615 <u>+</u> 30	300 <u>+</u> 15
4	5.30(D)	2.12	586 <u>+</u> 86	49 <u>+</u> 12	537 <u>+</u> 43	253 <u>+</u> 20
4	14.7(E)	4.20	734 <u>+</u> 46	49 <u>+</u> 12	685 <u>+</u> 23	163 <u>+</u> 5
4	C£-252(A	) 2.27	744 <u>+</u> 54	49 <u>+</u> 12	695 <u>+</u> 27	306 <u>+</u> 12
4	C£-252(B	) 2.02	658 <u>+</u> 77	49 <u>+</u> 12	609 <u>+</u> 38	301 <u>+</u> 19
4	C£-252(C	) 2.87	900 <u>+</u> 61	49 <u>+</u> 12	851 <u>+</u> 30	296 <u>+</u> 10
4	C£-252(D	) 1.99	632 <u>+</u> 26	49 <u>+</u> 12	583 <u>+</u> 13	293 <u>+</u> 7
4	C£-252(E	) 2.03	632 <u>+</u> 29	49 <u>+</u> 12	583 <u>+</u> 15	289 <u>+</u> 7
4	Thermel	0.0192 <sup>*</sup>	3870 <u>+</u> 470	49 <u>+</u> 12	3820 <u>+</u> 17	0 199000 <u>+</u> 9000

Irradiation at Harwell(A), Battelle(B), PTB(C), GSF(D), CRNL(E)

\* Separate irradiation carried out at AERE Harwell. CR39 exposed with BN1 boron foils.

No of samples	Neutron field or energy1) E (MeV)	Reference dose equivalent H (mSv)	Track density N± σ <sub>n-1</sub> (cm <sup>-2</sup> )	Background tracks N <sub>0</sub> ±σ <sub>n-1</sub> (cm <sup>-2</sup> )	Net track density N±SEM (cm <sup>-2</sup> )	Response R±SEM (cm-2·mSv-1)
4	0.144(C)	2.0	568 <u>+</u> 200	71 <u>+</u> 34	497 <u>+</u> 100	248 <u>+</u> 50
4	0.25(C)	1.73	1260 <u>+</u> 34	71 <u>+</u> 34	1189 <u>+</u> 17	687 <u>+</u> 10
4	0.57(D)	1.5	828 <u>+</u> 57	71 <u>+</u> 34	757 <u>+</u> 29	505 <u>+</u> 19
4	1.20(B)	2.09	960 <u>+</u> 50	71 <u>+</u> 34	889 <u>+</u> 25	425 <u>+</u> 12
4	2.85(E)	2.05	904 <u>+</u> 80	71 <u>+</u> 34	833 <u>+</u> 40	406 <u>+</u> 20
4	5.30(D)	2.12	752 <u>+</u> 104	71 <u>+</u> 34	681 <u>+</u> 52	321 <u>+</u> 24
4	14.7(E)	4.20	984 <u>+</u> 68	71 <u>+</u> 34	913 <u>+</u> 34	217 <u>+</u> 8

Viewing angle 20<sup>0</sup>

Irradiation at Harwell(A), Battelle(B), PTB(C), GSF(D), CRNL(E)

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European/US/Canadian Neutron Dosemeter Irradiations. Summary of Results from Risø National Laboratory.

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#### 1. Description of dosemeter

Two types of CR-39 were used: 1) Pershore grade PM355 (nominal thickness 500  $\mu$ m) produced by Pershore Mouldings Limited, UK, and 2) TASTRAK standard grade (nominal thickness 750  $\mu$ m) produced by Track Analysis Systems Limited, Bristol, UK. For both types, the material was manufactured in November 1985. Two sheets of each type were used. Following the irradiations in November-December 1986, the detectors were etched in January-March 1987. The material was stored throughout in air.

Each detector consisted of a 15 mm x 15 mm square. The detectors were placed in black polyethylene bags (100  $\mu$ m thick) and in addition they were covered by about 100  $\mu$ m of adhesive tape. They were read on the rear side, so the CR-39 itself acted as a proton radiator.

Each irradiation card (except the one irradiated with thermal neutrons) was equipped with fifteen CR-39 detectors of each type. The cards were provided with wedges allowing the detectors to be irradiated at five different angles of incidence:  $0^{\circ}$  (normal to neutron beam),  $15^{\circ}$ ,  $30^{\circ}$ ,  $45^{\circ}$ , and  $60^{\circ}$ . Hence, three detectors of each type were irradiated at each angle of incidence. For thermal neutrons, six detectors of each type were irradiated at normal incidence only. In this case the detectors were partly covered with LiF and Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> converters placed in contact with the front (3 detectors) or back (3 detectors) surface of the CR-39.

Nine background samples of each type accompanied the samples irradiated at each irradiation laboratory. Additional background samples were kept at Risø.

#### 2. Processing

The samples were etched in 6.25 N NaOH at 70°C for 16 hours.

#### 3. Assessment

The tracks on the rear side of the samples were counted using a semi-automated image analysis system composed of an optical microscope, CCD camera, frame grabber, personal computer and monitor (Ref. 1). When using this system several combinations of microscope condenser and objective may be chosen, so a variety of illumination conditions and magnifications may be used.

Initially it was found that the illumination conditions and grey-level threshold which had been used previously for counting of tracks from AmBe- or Cf-neutrons resulted in a sensitivity too low to detect the shallow tracks from 144 keV neutrons and 250 keV neutrons. Therefore, the system was adjusted to allow more shallow tracks to be counted, although this resulted in an increased background count on unexposed samples.

The results presented in table 1 were obtained by counting an area of 0.20  $\text{cm}^2$  for all samples.

#### 4. Results and discussion

The results obtained for the TASTRAK material for normal incidence are given in table 1. The reference dose equivalent values have been corrected for source-to-detector distance in accordance with the irradiation protocols provided by the irradiation laboratories. The result for thermal neutrons refers to the rear surfaces not covered with LiF and  $\text{Li}_2\text{B}_4\text{O}_7$  converters. Below LiF TLD-700 converters the response to thermal neutrons was  $901\pm55$  cm<sup>-2</sup> mSv<sup>-1</sup>. However, this value applies to the particular batch of LiF TLD-700 used here.

The results presented in table 1 show that an acceptable response as a function of neutron energy is obtainable for the whole neutron-energy range from 144 keV to 14.7 MeV. However, the sensitivity at 144 keV and 250 keV has been obtained at the expense of an increased background and an increased variability in background. For the two sheets of CR-39 used here, one standard deviation in background corresponds to about 0.24 and 0.30 mSv respectively, when reference is made to neutrons from Cf-252. The standard deviation in background is reduced to about 0.15 mSv if the track detection at 144 keV is suppressed, while maintaining the relatively high detected track density at 250 keV. This standard deviation is still higher than typical values obtained when using the higher intensity limit on the image analysis system used previously, or when using counting by eye. For these counting procedures, typical values of one standard deviation in background corresponds to 0.05 - 0.1 mSv for the image analysis system and below 0.05 mSv for counting by eye.

For the Pershore material used in this work an acceptable "signal-to noise" ratio has not been obtained at 144 keV and 250 keV with the illumination conditions used in the reading system so far. This seems to be because the material was without additives and so exhibited a poor contrast between shallow tracks and background. Above 570 keV, the relative response as a function of neutron energy was similar to that obtained for the TASTRAK material. The samples of both types will be further analysed with the aim of improving our system.

#### References

 Majborn, B. Automated Counting and Analysis of Etched Tracks in CR-39 Plastic. Radiat. Prot. Dosim. <u>17</u>, 165-169 (1986).

CR-39 sheet code	No of samples	Neutron field or energy1) E (MeV)	Reference dose equivalent H (mSv)	Track density N±σ <sub>n-1</sub> (cm <sup>-2</sup> )	Background tracks N <sub>0</sub> ± σ <sub>n-1</sub> (cm-2)	Net track density N±SEM (cm <sup>-2</sup> )	Response R±SEM (cm-2·mSv-1)
 Tl	3	0.144 (C)	2.27	811+183	334+ 85	477+107	210+ 47
TF	3	0.250 (C)	1.71	1483+147	11	1149+ 86	672+ 50
11	3	0.57 (D)	1.50	_ 1118 <u>+</u> 145	25	- 784 <u>+</u> 85	_ 523+ 57
e1	3	1.2 (B)	2.09	1184 <u>+</u> 77	F1	850 <u>+</u> 47	407 <u>+</u> 22
11	3	2.85 (E)	2.09	1108 <u>+</u> 106	11	774 <u>+</u> 63	370 <u>+</u> 30
79	3	5.30 (D)	2.12	924+ 96	97	590+ 57	278+ 27
15	3	14.71 (E)	4.29	913 <u>+</u> 67	77	- 579 <u>+</u> 41	- 135 <u>+</u> 10
Т2	3	Cf-252 (A)	2.18	1310 <u>+</u> 80	512 <u>+</u> 106	798 <u>+</u> 50	366 <u>+</u> 23
17	3	Cf-252 (B)	2.02	1299 <u>+</u> 125	IJ	787 <u>+</u> 75	390 <u>+</u> 37
19	3	Cf-252 (C)	2.20	1166 <u>+</u> 76	11	654 <u>+</u> 48	297 <u>+</u> 22
11	3	Cf-252 (D)	1.99	1186 <u>+</u> 77	11	674 <u>+</u> 49	339 <u>+</u> 25
19	3	Cf-252 (E)	2.05	1266 <u>+</u> 78	11	754 <u>+</u> 49	368 <u>+</u> 24
11	3	thermal (A)	2,65 <sup>2)</sup>	616+ 74	17	104+ 47	(nil) <sup>3)</sup>

Table 1. Results of CR-39 exposures (normal incidence).

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

2) Accompanying fast-neutron dose equivalent is  $\sim$  0.26 mSv.

3) After subtraction of contribution due to accompanying fast neutrons.

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#### CENDOS NEUTRON IRRADIATIONS OF KODAK CN-85

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1. DESCRIPTION OF DOSEMETER.

CN-85 cellulose nitrate (100  $\mu m$  thickness) manufactured by Kodak-Pathé France has been irradiated in the framework of CENDOS.

Dosemeters were covered by 1mm of polyethylene on each side during irradiation (detection of protons). A background control dosemeter accompanied the various series of dosemeters sent to different institutions. No special sealing was used for dosemeters packing.

The Kodak CN-85 used in this work was obtained from the manufacturer in august 1986 and stored in a refrigerator until use. Dosemeters were prepared in october 1986 and sent to 5 laboratories. After irradiation by different sources, they were stored in a refrigerator in contact with the polyethylene convertor until processing which began in march 1987.

2. PROCESSING.

Detector processing consists of an electrochemical etching :

. 3.5 hours at 50°C
. NaOH (5N) solution
. Electric field : 15 kV.cm<sup>-1</sup> rms.
. Frequency : 5 kHz.

We used a device constructed in the laboratory generating a sinusoidal signal (0 - 10 kHz; 0-400 volts rms; 10 mA max).

The cell used enabled only 4 dosemeters to be etched at a time; all detectors were processed under the same conditions in the same cell. For each etching, we treated in the cell 1 (or 2) background foils and 3 (or 2) detectors irradiated at different energies.

The electric field was adjusted after measuring the thickness of the different CN-85 foils. The dosemeters were etched on one side only (irradiated side); under these conditions the removed layer was 5.6  $\pm$  0.8  $\mu$ m (mean of 57 evaluations).

#### 3. ASSESSMENT.

A circular area of about 1  $cm^2$  is electrochemically etched.

An area of about 0.6  $\text{cm}^2$  was evaluated, this represents about 75 fields. We used an optical microscope (LEITZ) at a magnification of x 100. All detectors were evaluated by simple eye-counting.

Under our etching conditions, spots with a mean diameter 30  $\mu m$  (maximum 45  $\mu m$ ) are obtained.

#### 4. RESULTS.

The results obtained are given in the attached table. They correspond to normal incidence of the neutron beam on the detector.

For the background evaluation, we took the mean of all the samples (21) etched together with the different irradiated dosemeters.

The standard deviations of the different groups of results were calculated even for relatively small samples.

No of samples	Neutron field or energy <sup>1)</sup>	Reference dose equivalent	Track density N $\pm \sigma_{n-1}$	Background tracks No± σ <sub>n-1</sub>	Net track density $N \pm SEM$	Response R±SEM
	L (IMEV)		(CIII-2)	(CIII-2)	(CIII-2)	(CIII-2-IIISV-1)
3	0.144 (C)	1.97	$220.5 \pm 12.6$	194.9 <u>+</u> 42.8	25.6 + 11.8	13.0 <u>+</u> 6.0
3	0.250 (C)	1.80	347.6 <u>+</u> 30.6	11	152.7 <u>+</u> 20.0	84.8 <u>+</u> 11.1
2	0.565 (D)	1.50	512.9 <u>+</u> 40.5		318.0 <u>+</u> 30.1	212.0 <u>+</u> 20.1
3	1.200 (B)	2.09	477.2 + 22.3	11	282.3 <u>+</u> 15.9	135.1 <u>+</u> 7.6
3	2.800 (E)	2.05	285.7 <u>+</u> 25.8	11	90.8 <u>+</u> 17.6	44.3 <u>+</u> 8.6
2	5.300 (D)	2.12	321.2 + 8.5	11	126.3 <u>+</u> 11.1	59.6 <u>+</u> 5.2
3	14.700 (E)	4.20	453.6 <u>+</u> 63.2	11	258.7 <u>+</u> 37.7	61.6 <u>+</u> 9.0
3	Cf-252 (A)	2.27	533.2 <u>+</u> 45.7	11	338.3 <u>+</u> 28.0	149.0 <u>+</u> 12.3
3	Cf-252 (B)	2.02	449.6 + 47.8	n	254.7 <u>+</u> 29.1	126.1 <u>+</u> 14.4
3	Cf-252 (C)	2.87	449.1 <u>+</u> 65.1	TI	254.2 <u>+</u> 38.7	88.6 <u>+</u> 13.5
2	Cf-252 (D)	1.99	387.4 <u>+</u> 40.2	11	192.5 <u>+</u> 29.9	96.7 <u>+</u> 15.0
3	Cf-252 (E)	2.03	374.5 + 54.7	IT	179.6 <u>+</u> 32.9	88.5 <u>+</u> 16.2
2	thormol (A)	(2.37)	<u>881 0 ± 69 0</u>	11	(755.6)	(319) 298.9 + 15.6
د ١	mermai (A)	2.05			,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	

<sup>1)</sup> Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

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At present we are studying with Kodak a new product of same composition as CN-85 but with a thickness of 200  $\mu$ m. With such a thickness we will etch the dosemeter for a longer time and in this way we hope to increase the sensitivity without risk of etched tracks piercing the film. We need a product with a good surface state; joint studies with Kodak are underway.

We are also developing a new cell system capable of etching 24 or more detectors if possible (depending on the power of high voltage source) in order to resolve routine dosemetry problems.

## SUMMARY OF RESULTS OBTAINED AT PACIFIC NORTHWEST LABORATORY FROM JOINT NEUTRON IRRADIATIONS OF NEUTRON TRACK DETECTORS

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## 1. <u>Description of Dosimeters</u>

The CR-39 material used in these exposures was made by PPG, Industries and cast by American Acrylics in three separate batches identified as batches C, D, and E. Samples from batch C were made from 94% pure monomer and covered front and back with 0.13 mm of polyethylene. Samples from batch D were made from 85% monomer distilled to about 98% purity. This batch has a high neutron sensitivity but suffers from a higher than usual background partly, perhaps, because its polyethylene cover is only 0.06 mm thick. Batch E was made from 94% pure monomer and also has a thin 0.06 mm polyethylene covering. Six dosimeters from each batch were mounted on the sample cards. Although double wrapped in plastic during travel, no additional radiator material was used during irradiations.

## 2. Processing

The large number of samples from three separate batches were separated for processing to analyze the differences in the energy response for three different processing methods. Three samples from each card exposed to monoenergetic neutrons and six samples from the Cf-252 exposures were set aside for electrochemical etching using the following parameters:

> Cycle 1 - 39 kV/cm, 60 Hz, 60°C, 5 h Cycle 2 - 39 kV/cm, 2 kHz, 60°C, 23 min

The remaining samples were processed using chemical etching alone or in a combination of the methods and parameters and are not reported here.

<sup>\*</sup> Operated by Battelle Memorial Institute for the U.S. Department of Energy under contract DE-AC06-76RL0 1830.

#### 3. Assessment

The sample track densities were analyzed using an optical microscope and an image analysis system that automatically counts the tracks and can calculate a track size distribution. About 0.53 cm<sup>2</sup> of surface was scanned per sample. Track size distributions were analyzed using an interval step size of 2.5 X  $10^{-6}$  cm<sup>2</sup>.

## 4. Discussion of Results

A summary of the results of this analysis shows a net response from 189 to 992 tracks/ $cm^2$ -Sv (see Table 1). Samples from all energies except for 0.565 and 5.300 MeV were run simultaneously. The 0.565 and 5.30 MeV samples were processed later using the same parameters. The dosimeter response to thermal radiation was within one standard deviation of the background average and assumed to be zero. The polyethylene radiator in front of the dosimeters was not thick enough to block detection of proton contamination in the 14.7 MeV beam. Using the factors supplied by Bill Cross, Chalk River National Laboratory (CRNL), the track response was corrected to represent only the neutron-induced proton recoils. Track size analysis resulted in the size distribution histograms shown in Figures 1 and 2. The shapes rather than the magnitude are of most interest. The exact histogram shape depends largely on interval size. These graphs are based on the total track numbers over the area scanned of one sample from each exposure and are not normalized to account for the difference in fluence.

The Cf-252 exposures were performed at Harwell, Pacific Northwest Laboratory (PNL) (operated by Battelle Memorial Institute), Physikalisch-Technische Bundesanstalt (PTB), CRNL, and Gesellschaft für Strahlen- und Umweltforschung (GSF) irradiation facilities using samples from batches C, D, and E. Results of these three runs have been averaged together to give composite readings. These three batches have slightly different response sensitivities and show a higher variation than samples from a single batch. According to these averages, PNL is on the high side of average, Harwell is on the low side, and the remaining groups are in between.

Probably the most exciting result from this analysis is the differences in track size distributions of the various monoenergetic neutrons and their conspicuous differences, in some cases, from the Cf-252 distributions. The value of size distribution analysis will be further investigated. A number of problems with analysis of personnel dosimetry using track size to evaluate energies of exposure come to mind immediately, including personnel exposed to a combination of sources as well as insufficient or excessive track densities, changes in processing parameters, and discriminating dissimilar spectra from similar distributions, e.g., 144 and 2800 keV. However, this technique may prove useful in certain situations and its study will be pursued.



FIGURE 1. Track Size Distributions of CR-39 Exposed to Cf-252 Sources from Various Irradiation Facilities (not normalized for fluence)



FIGURE 2. Track Size Distributions for the Indicated Neutron Energies (not normalized for fluence)

TABLE 1.	Results	of CR-39	Exposures

No of	Neutron field or energy1)		Reference dose equivalent	Track density	Background tracks	Net track density	Response
samples	E (MeV)		H (mSv)	$\frac{N \pm \sigma_{n-1}}{(cm^{-2})}$	$\begin{array}{c} N_0 \pm \sigma_{n-1} \\ (cm^{-2}) \end{array}$	$\frac{N \pm SEM}{(cm^{-2})}$	R±SEM (cm <sup>-2</sup> ·mSv <sup>-1</sup> )
3	0.144 (	C)	2.0	1369± 77.1	174±33.0	1195± 44.5	598±22.2
3	0.250 (	C)	1.73	1890±190.7	174±33.0	1716±110.1	992±65.6
2	0.565 (	D)	1.50	1348± 11.3	174±33.0	1174± 8.0	783± 4.0
3	1.200 (	B)	2.09	1712± 70.7	174±33.0	1538± 40.8	736±21.1
3	2.800 (	E)	2.05	1353± 28.6	174±33.0	1179± 16.5	575± 8.1
2	5.300 (	D)	2.12	574± 9.2	174±33.0	400± 6.5	139± 3.5
3	14.700 (	E)	4.20	1144±208 <sup>(a)</sup>	174±33.0	969 <u>+</u> 120.1	231±18.4
						(1.)	(6)
6	Cf-252 (.	A)	2.27	1708±250.9 <sup>(b)</sup>	203±16.4	1505±144.9 <sup>(D)</sup>	663±45.3 <sup>(D)</sup>
6	Cf-252 (	B)	2.022	1723±347.9 <sup>(b)</sup>	203±16.4	1520±200.9	752±90.3
6	Cf-252 (	C)	2.87	2265±211.1 <sup>(b)</sup>	203±16.4	2062±121.8	718±42.6
4	Cf-252 (	D)	1.994	1684±295.2 <sup>(b)</sup>	203±16.4	1481±170.4	743±85.4
6	Cf-252 (	E)	2.03	1681±227.0 <sup>(b)</sup>	203±16.4	1478±131.1	728±64.6
3	thermal (	A)	2.63	181± 24.6	174±33.0	7.0± 14.6	-0-

1) Irradiation at Harwell (A), Battelle (B), PTB (C), GSF (D), CRNL (E)

- (a) corrected for proton contamination.
- (b) mean represents results of 3 separate CR-39 batches with different neutron sensitivities, actual percent deviation of each batch ran 0.4-6% for 98% pure batch, 3-12% on 94% pure batches.

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## <u>Conclusions</u>

The purpose of these joint irradiations was to give participants an opportunity to expose their detectors to known neutron doses for a wide range of energies, and to give a general indication of the relative performance of all the systems under equivalent irradiation conditions.

In the first programme of joint neutron irradiations, which took place in 1984 (see Harwell Report AERE-R11926), eight European groups participated, submitting eleven systems, of which five were based on chemical etching (CE) and six on electrochemical etching (ECE). Three systems (two CE and one ECE) used automated counting methods. In this (the second) programme, a total of thirteen groups participated, including two from the USA and one from Canada, and sixteen systems were submitted. (Only fifteen systems are reported here, as one European group declined to report their results). Of these fifteen systems, five are CE systems and ten are ECE systems. Of the five CE systems, two used automated counting and three used counting by eye; five of the ten ECE systems used automated counting and five used counting by eye.

Because of the developmental nature of these trials, it would be unwise to attempt to draw any final conclusions from the results presented in the reports, but the data assembled in Table 1 is intended to give some idea of the relative performance of each system at its present state of development. In columns (a) and (b) the type of system (CE or ECE) and the counting method employed (automatic or eye) are indicated. Column (c) gives the response (track or ECE spot density) for normally-incident Cf fission neutrons - the unweighted mean of the results obtained from exposures at the five irradiation laboratories. Column (d) gives the per cent fractional standard deviation of these five measurements to indicate variability of the system (assuming that all Cf doses were accurate). Columns (e) and (f), give the ratio of the dose-equivalent response for 140 keV neutrons to the Cf neutron response, and the ratio of the dose-equivalent response for 14.7 MeV neutrons to the Cf neutron response, respectively; thus, these give an idea of the energy-response of each system. Finally, columns (g) and (h) give the background, and variations in the background, in terms of the dose-equivalent deduced from the response to Cf neutrons, respectively. These give some idea of the limits of detection of the systems. It should be noted that all these data refer to normally-incident neutrons only, and any operational dosimetry system would have to allow for the angular distribution of neutrons incident on the dosimeter. This problem has not been considered in these joint irradiations.

From this Table it is evident that all systems have good sensitivity, and most have an acceptable energy response. The responses of individual detectors show, in some cases, considerable variability. Some systems have background uncertainties below ~0.1 mSv and are thus close to being acceptable for operational use: any further conclusions are left to the reader.

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	Etching System (CE or ECE)	Counting System (eye or auto)	Mean response to Cf fission neutrons	SD of Cf responses as measured at five laboratories as a fraction of mean response	Ratio of dose- equivalent response to 140 keV neutrons compared with Cf fission	Ratio of dose- equivalent response to 14.7 MeV neutrons compared with Cf fission	Background in terms of Cf dose- equivalent	l SD on background in terms of Cf dose- equivalent
			0 1	-	neutrons	neutrons		
			$(cm^{-2} mSv^{-1})$	(%)			(mSv)	(mSv)
	(a)	(b)	(c)	(d)	(e)	(f)	(g)	(h)
LA	ECE	eye	555	12	1.93	0.40	0.58	0.06
LB	ECE	eye	452	15	3.34	0.42	0.44	0.23
2	ECE	auto	160	9	0.40	0.46	0.64	0.11
3	ECE	auto	43	19	1.40	1.08	0.30	0.12
μÂ	CE	eye	969	11	0.44	0.41	0.67	0.19
μB	ECE	auto	426	21	2.29	0.45	3.03	0.40
5	EXE	eye	594	17	1.07	0.72	0.05	0.02
5A	CE	eye	636	-	0.64	-	1.89	0.61
5B*	CE	eye	529	-	1.21	-	2.38	0.59
7	ECE	eye	107	12	1.12	0.50	0.19	0.05
3	ECE	auto	424	9	0.73	0.53	0.12	0.04
9	CE	auto	297	2	0.0	0.55	0.16	0.04
L0	CE	auto	352	10	0.60	0.38	1.45	0.30
11+	ECE	eye	110	24	0.12	0.56	1.77	0.39
12	ECE	auto	721	5	0.83	0.32	0.28	0.02

## Table 1Summary of selected results from the participants to give an indication of sensitivity,<br/>energy response and limit of detection for each system

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