

The Henryk Niewodniczański  
INSTITUTE OF NUCLEAR PHYSICS  
Polish Academy of Sciences  
152 Radzikowskiego Str., 31-342 Kraków, Poland

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Kraków, August 2005

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Report No. 1964/D

Dosimetric properties of the TLD European Cards with high sensitive  
MCP-7 ( ${}^7\text{LiF:Mg,Cu,P}$ ) thermoluminescence detectors for personal  
dosimetry

**Paweł Bilski and Maciej Budzanowski**

***Abstract***

Dosimetric properties of Harshaw compatible cards equipped with two thermoluminescence MCP-7 (LiF:Mg,Cu,P) detectors were investigated at the accredited Laboratory of Individual and Environmental Dosimetry (Polish acronym LADIS) at the Institute of Nuclear Physics, Krakow. Dose detection threshold, dose sensitivity, dose linearity, residual signal, fading, sensitivity to thermal neutrons, sensitivity to ambient light, and the influence of environmental conditions were evaluated. All investigated dosimetric properties were found to be within the limits defined by IEC document 1066 "Thermoluminescence Dosimetry Systems for Personal and Environmental Standard".

## INTRODUCTION

There is an increasing interest among the dosimetric services in Europe to replace the outdated film dosimeters with the modern personal dosimeters e.g. with high sensitive thermoluminescence detectors (TLD). In 2004 Radcard/TLD Poland developed the new TLD European Card (TLD-EC), with lithium fluoride TLD detectors, fully compatible with the Harshaw/Bicron dosimetric cards.

The aim of this report is to evaluate the selected dosimetric characteristics of TLD-EC cards equipped with two thermoluminescence MCP-7 (LiF:Mg,Cu,P) detectors with diameter 3.6 mm and thickness 0.38 mm and 0.26 mm placed in positions 2 and 3. The report is based on results of investigations performed at the Institute of Nuclear Physics (IFJ), Krakow in Laboratory of Individual and Environmental Dosimetry (Polish acronym LADIS). The irradiations were performed at the Laboratory for Calibration of Radiation Protection Instruments (Polish acronym LWPD). LADIS and LWPD are accredited at the Polish Centre of Accreditation, PCA, under the numbers A049 and A029, respectively. Some conclusions on the dosimetric characteristics of the TLD-EC were drawn basing on the results of earlier studies on the dosimetric properties of MCP-7 detectors.

## MATERIAL AND METHODS

### -TLD European Card

TLD European Card (TLD-EC) was developed in 2004 by Radcard/ TLD Poland. Several examples of the cards in different colors are presented in Fig. 1. In the card two types of lithium fluoride thermoluminescence detectors are installed: standard LiF:Mg,Ti (MTS) or high sensitive LiF:Mg,Cu,P (MCP) detectors developed at IFJ Krakow and produced by TLD Poland. Both MCP and MTS detectors are available with natural lithium isotope content (MCP-N, MTS-N), with enriched Li-7 (MCP-7, MTS-7) and enriched Li-6 (MCP-6, MTS-6). Up to four TLD detectors, in form of round pellets (typical diameter is 3.6 mm) or square chips (3.2 mm x 3.2 mm) can be placed in the card. The thickness of the detectors is 0.38 mm or less, if requested.

All investigations were performed with 50 TLD-EC cards, equipped with two MCP-7 detectors, diameter 3.6 mm in diameter and thickness 0.38 mm in position 2 and 0.26 mm in position 3 were applied.

### - TLD reader

All cards were evaluated on Harshaw reader model 8800 kindly provided for these studies by SynOdys Passive Dosimetry.

For readout the nitrogen of class 04 with purity 99.99% was applied.

The following Time Temperature Profile (TTP) was applied for readout of both TLDs:

- Preheating: 170°C, 10 s
- Heating (linear): 25°C/s up to 250°C
- Acquisition time: 20 s

Standard voltage applied during the readout was 800 V for all four photomultipliers



Fig. 1. TLD European Card (TLD-EC) with 2 MCP-7 detectors in positions 2 and 3

#### - Irradiation facilities

The  $\gamma$ -ray irradiation was performed at the calibration facility of LRPD laboratory. The irradiation room is located in a hall of dimension 9 m (length) x 4 m (wide) x 4.5 m (height). For calibration purposes the  $^{137}\text{Cs}$  source of activity 185 MBq (5 Ci) was applied. This source covers a dose rate range from 290  $\mu\text{Gy/h}$  to 17 mGy/h. The calibrated dosimeters are placed on a vehicle remotely positioned at the distance from the source between 1 and 7 meters. For high dose irradiation the  $^{137}\text{Cs}$  source at the cyclotron building (room 0011) was applied. UNIDOS dosimeter with PTW ionisation chambers is used for determination of the dose rate.

Part of exposures was performed with the internal  $^{90}\text{Sr}/^{90}\text{Y}$  irradiator of the Harshaw/Bicron 8800 reader, which was previously calibrated by LRPD laboratory.

The absolute measurements of MCP-7 detectors sensitivity to thermal neutrons were performed in 2004 at the Geesthacht Neutron Facility operated by PTB Braunschweig.

#### - Quantities

The requirements and tests presented within this report are expressed in terms of conventional true value of dose,  $D$  and evaluated dose  $E$ , according to the convention used in document IEC 1066. For personal dosimetry both quantities are intended to give the dose equivalent at the 10 mm depth in tissue.

## RESULTS

### 1) Detection threshold

n=40 non-irradiated cards were prepared and read out. For each card the value  $E$  for each unirradiated card was evaluated, the mean values  $\bar{E}$  and standard deviation  $s_{\bar{E}}$  for all 40 cards. Detection threshold  $D_0$  is defined as

$$D_0 = t_n s_{\bar{E}} \quad (1)$$

In Fig. 2 the distribution of measured dose,  $E$  for unirradiated cards is presented for detectors 0.38 mm (in position 2) and the detector 0.26 mm (in position 3). The calculated  $s_{\bar{E}}$  were equal 0.48  $\mu\text{Sv}$  and 0.83  $\mu\text{Sv}$ . Taking from the Student distribution  $t_{40} = 2.02$  the corresponding detection thresholds were equal:

$D_0 = 0.97 \mu\text{Sv}$  for the MCP-7 detector 0.38 mm and  
 $D_0 = 1.68 \mu\text{Sv}$  the MCP-7 detector for 0.26 mm.

According to the requirements formulated in IEC 1006  $D_0$  should not exceed 100  $\mu\text{Sv}$ .

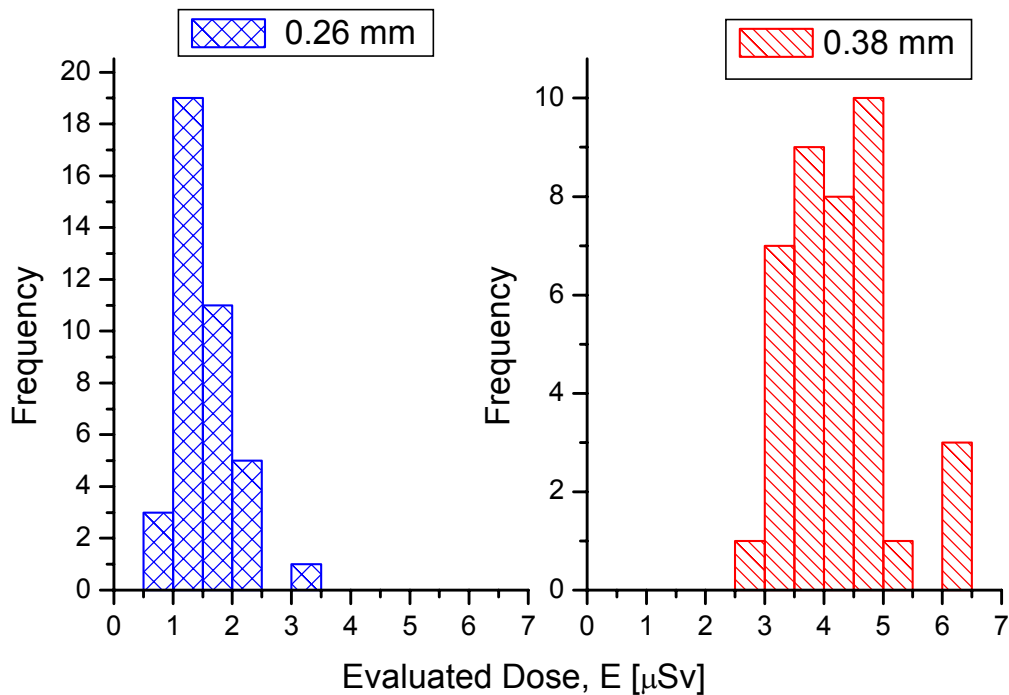


Fig. 2. Distribution of evaluated dose for read out of unirradiated TLD-EC with 0.38 mm and 0.26 mm thick MCP-7 detectors.

## 2) Dose sensitivity

Dose sensitivity,  $C$ , defined as conversion factor from the collected charge (in nC) to dose (in mSv), is a highly non-universal parameter. It depends strongly on the type of TLD reader and its settings. In particular, the increase the photomultiplier voltage significantly increases the dose sensitivity value.

For the particular 8800 reader, which was exploited during reported tests, with its standard settings (800 V photomultiplier tube), following values were measured:

$$C = 338 \text{ nC/mSv (position 2)}$$

$$C = 224 \text{ nC/mSv (position 3)}$$

For readout of very high doses (up to 20 Gy) the voltage on all photomultipliers was decreased to 500 V. Then, the sensitivity factors were equal:

$$C = 10.3 \text{ nC/mSv (position 2)}$$

$$C = 6.87 \text{ nC/mSv (position 3)}$$

For the measurements performed using the reader model 6600, the corresponding values were reported [Bud2005]:

$$C = 509 \text{ nC/mSv (position 2)}$$

$$C = 309 \text{ nC/mSv (position 3)}$$

## 3) Dose linearity

Irradiations necessary to test linearity of dose response were performed with  $^{137}\text{Cs}$  source at the accredited Laboratory for Calibration of Radiation Protection Instruments (LWPD) at the IFJ. Part of exposures was performed with the internal  $^{90}\text{Sr}/^{90}\text{Y}$  irradiator of the 8800 reader, which was previously calibrated at the LWPD laboratory. The current dose response studies (triangles, Fig. 3) were performed for doses between 100  $\mu\text{Gy}$  to 20 Gy. According to the IEC1066 the measured response shall not vary from the conventional true value,  $D$ , by not more than 10% over the following range. The results are presented in Table 1.

Table 1.

Evaluation of the linearity of response of TLD-EC cards irradiated with  $\gamma$ -rays from  $^{137}\text{Cs}$  source.  $n = 4$  cards were irradiated at each dose level.

Conventional true dose, $D_i$ [mSv]	$I_i = \frac{t_{ni} s_i}{\sqrt{n_i}}$	$\frac{\overline{E}_i \pm I_i}{D_i}$
0.1	0.035	(0.95, 1.02)
1	0.040	(0.96, 1.04)
10	0.035	(0.96, 1.04)
100	0.023	(1.00, 1.05)
1000	0.022	(0.98, 1.02)

The results are compared with earlier studies, performed for bare MCP-N detectors with  $^{137}\text{Cs}$  and  $^{60}\text{Co}$  radiation (see Figure 2). The solid lines in the figure represent 10% band around the trend of perfect linearity. According to the IEC1066 standard the response is treated as linear if it keeps within these limits. It should be however mentioned, that while the IEC1066 puts

the requirement of linearity up to dose of 1 Gy only, the tested dosimeters fulfil this requirement up to 20 Gy. Whats more, the response is linear within 5% up to at least 10 Gy.

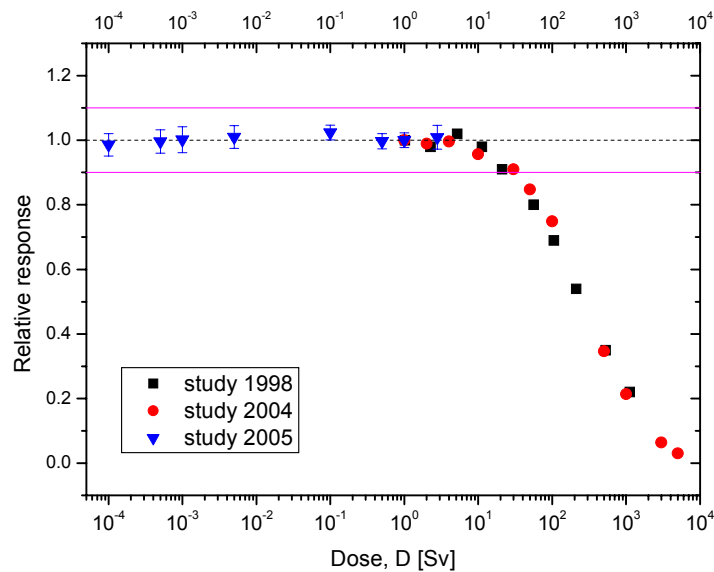


Fig. 3 Dose response of MCP detectors – the new results compared with those of earlier studies. For “Study 1998” [Bil1998] irradiations were performed with  $^{137}\text{Cs}$  radiation at the IFJ, for “study 2004” [Olk2004] irradiations were performed with  $^{60}\text{Co}$  radiation at KAERI (Korea).

#### 4) Residual signal

Residual signal is a signal remaining in the TL detector after the readout. The value of the residual signal is determined by the subsequent read out of the detector without prior irradiation and usually without additional annealing.

To test the residual signal, TLD-EC cards were exposed to doses of 0.7, 50, 100, 500, and 1000 mSv by  $^{90}\text{Sr}$  radiation (4 cards at each dose level) and read out with the standard TTP. Immediately afterwards all cards were re-read twice in the same conditions, to measure the residual signal. The results, expressed as the average percentage of the initial signal left in the TL detectors, are presented in the Table. 2

Table 2 .

Residual signal, expressed as an average percentage of the initial signal, measured within two consecutive re-readouts following a readout of exposed detectors.

	Position 2					Position 3				
Dose, [mSv]	0.7	50	100	500	1000	0.7	50	100	500	1000
1 <sup>st</sup> re-read	1.33%	1.28%	1.15%	1.08%	1.05%	1.14%	0.80%	0.83%	0.89%	0.64%
2 <sup>nd</sup> re-read	-	0.54%	0.47%	0.44%	0.43%	-	0.31%	0.32%	0.36%	0.25%

It can be seen that the residual signal after first readout is at the level of 1%. The next readout, which can be treated as a simplified annealing, reduces it below 0.5%. Such second readout performed if signal exceeds a set limit may be easily implemented in the readout procedure if necessary (reader software enables such option). The observed difference between TLDs in position 2 and 3 is caused by different thickness of both detectors. The thinner detector in position 3 heats up quicker and therefore remains longer at the end temperature of 250°C, what reduces the residual signal.

For low dose irradiation, the measured signal includes also the non-radiation background. For residual signal measured after 0.7 mSv irradiation (which expressed in dos units was equal to 9.3 µSv), about 50% of signal is of the non-radiation origin (see Fig. 1).

To fulfil the requirements of IEC 1066 for residue after dose of 100 mSv, a reader annealing of LiF:Mg,Cu,P detectors is recommended.

Another measurement of the residual signal was performed during the described below reproducibility study. Ten cards underwent a series of irradiation (1 mSv) and readout cycles. After each ten cycles an additional readout was performed to remove any signal remaining in the detectors. In this way an accumulated residual of ten irradiations was measured. The obtained values were quite low: 0.27% (position 2) and 0.18% (position 3).

### **5) Time effects - fading**

During storage all thermoluminescent materials show some instability of the response (fading) which is a function of time and storage temperature. Fading is an intrinsic effect of a TL material and encapsulation of TL elements into a dosimetric card has no influence.

The problem of fading of MCP-N (LiF:Mg,Cu,P) detectors was thoroughly studied at IFJ Krakow in collaboration with CIEMAT (Madrid), within a project focused on application of this material in environmental dosimetry. Results of measurements, which were performed using MCP-N detectors produced by TLD Niewiadomski company (former name of TLD Poland), were published in a series of scientific papers [Bud1998, 1999, 2002, Sae1999, Gom1998].

In Fig. 4a the relative TL intensity of MCP-N (LiF:Mg,Cu,P) detectors, irradiated with  $\gamma$ -rays (10 mSv) versus time elapsed from irradiation is presented. The most important conclusion from these studies is that no decrease of the main dosimetric peak (number 4) was observed. This is true not only for storage of TLDs at room temperature, but also at elevated temperature of +50°C. Time period of all these investigations was limited to 3 months, but very good stability of this peak at elevated temperature, ensures that even if TLDs are stored for longer time at room temperature, effects of fading will be negligible. Peak number 3 (which occurs between 170°C-210°C, depending on heating rate) is obviously much less stable. After 3 months storage at room temperature only a few percent of the initial area of this peak is left. However this initial area constitutes only about 10% of the main peak area. This numbers were found via deconvolution of glow-curves measured with linear heating ramp without any pre-heat neither in reader nor in oven. In practice, when a reader pre-heat at temperatures about 160-175 °C is a standard procedure, contribution of this peak to the total signal will be greatly reduced.

The longest fading studies with MCP-N detectors were performed at the low-radiation level PTB underground laboratory LIBO/Asse near Braunschweig, Germany. MCP-N detectors

exposed at PTB and stored 1.5 year at the depth of about 1000 m in temperature 33°C lost about 5% of the signal [Bud1996].

Summarizing all these results, one may draw a conclusion, that fading of MCP-7 total signal during 6 months storage will be not greater than a few percent.

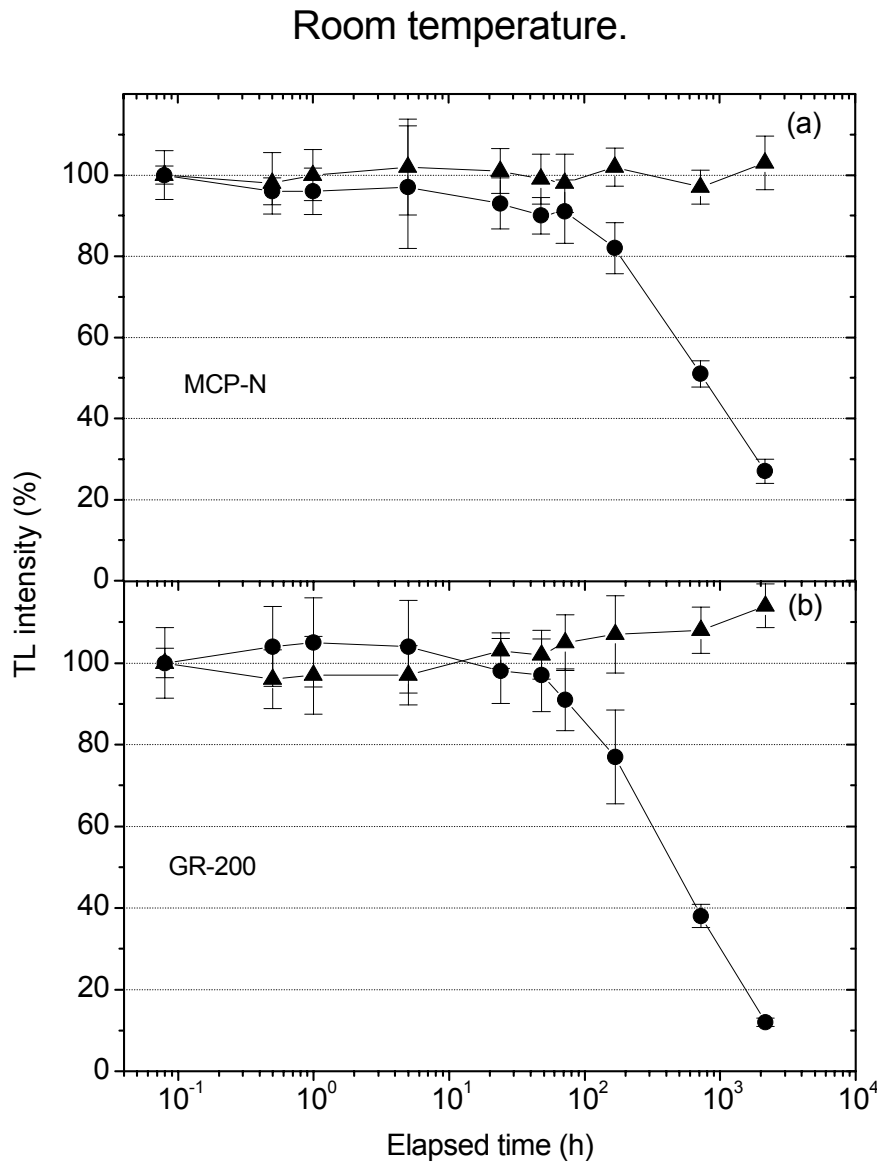


Fig. 4  
Relative TL intensity of MCP-N (LiF:Mg,Cu,P) and Chinese GR-200 (LiF:Mg,Cu,P) detectors, irradiated with  $\gamma$ -rays (10 mSv) versus time elapsed from irradiation. Circles represent peak No. 3 and Triangles the peak No. 4 (main dosimetric peak of LiF:Mg,Cu,P (Bud 1999))

### 6) Sensitivity to neutrons

Sensitivity of thermoluminescent cards to neutrons is determined by sensitivity of TL elements. In case of detectors based on lithium, like LiF:Mg,Cu,P, the most important is role of  $^6\text{Li}$  isotope, which has a very high cross-section for reaction with thermal neutrons (for



higher energies the cross-section decreases like  $1/v$ , see Fig. 5) and which traces are always present even in highly enriched  $^7\text{Li}$ . Encapsulation of TL elements in a dosimetric card has no effect on the neutron sensitivity; therefore results obtained for loose elements are still valid for cards.

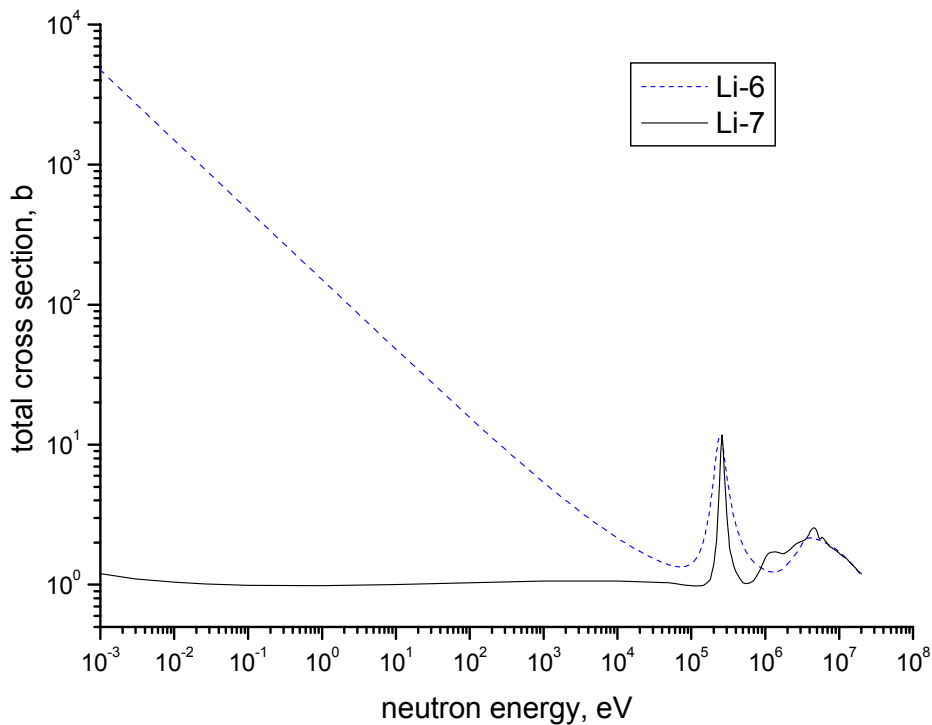


Figure 5. Microscopic cross-section of  $^6\text{Li}$  and  $^7\text{Li}$  for nuclear reactions with neutrons

A precise measurement of MCP-7 sensitivity to thermal neutrons has been recently performed by Burgkhardt *et al* [Bur2004] within a study on application of TLDs to photon dose measurements in Boron Neutron Capture Therapy, BNCT. In frame of these investigations, different TLDs were exposed to the reference thermal neutron field operated by PTB Germany at the Geesthacht Neutron Facility (GeNF). This is practically pure thermal neutron beam, characterized by very low gamma intensity and negligible fraction of epithermal neutrons. Among tested lithium based TL detectors, MCP-7 produced by TLD Poland exhibited the lowest response to thermal neutrons,  $R_{MCP-7}$ :

$$R_{MCP-7} = 1.3 \cdot 10^{-9} \text{ mGy}/(\text{neutr}/\text{cm}^2).$$

The meaning of  $R_{MCP-7}$  is, that the fluence of  $10^9 \text{ neutr cm}^2$  is needed to obtain TL signal of intensity equal to that produced by 1 mGy of gamma radiation. The value measured for MCP-7 is equal to 46% of TLD-700 response and 0.6% of TLD-100 response (Bur2004). The lower response of LiF:Mg,Cu,P (MCP-7) then LiF:Mg,Ti (TLD-700) to neutrons is a result of lower efficiency of this material to alpha particles and tritons, produced in  $n(^6\text{Li}, \alpha)\text{T}$  reaction [Bil1994]. It should be also noted that most of neutron fields in radiation protection practice is not fully thermalized, therefore actual neutron sensitivity of MCP-7 will be even lower.

## 7. Sensitivity to ambient light

There are two effects observed for thermoluminescence detectors related to the exposure to ambient light:

**a) *Effect on zero point i.e. the increase of TLD signal background signal, observed in the non-irradiated detectors***

The first effect is connected with the excitation and trapping of charge carriers at shallow and thermally unstable traps. The effect can take place in TLD material and in the materials of the holder. This signal is usually removed during the pre-heat at temperatures much below the main dosimetric peak.

Light sensitivity was tested in following way. Two groups of 3 cards have been annealed in the reader. Three of them were left in a standard laboratory light, without any cover and three of them were stored in darkness. After 24 hours the cards were read out using standard TTP. The obtained signals, expressed using dose units, were following:

Position 2: D= 9.4  $\mu$ Sv  
Position 3: D= 12.4  $\mu$ Sv

**b) *Effect on response i.e. the decrease of TLD signal after exposure to light of irradiated detectors***

The decrease of TLD signal after exposure to ambient light is due to bleaching i.e. removing the charge from some high-temperature traps after exposure to light, essentially to light with some contribution of UV component.

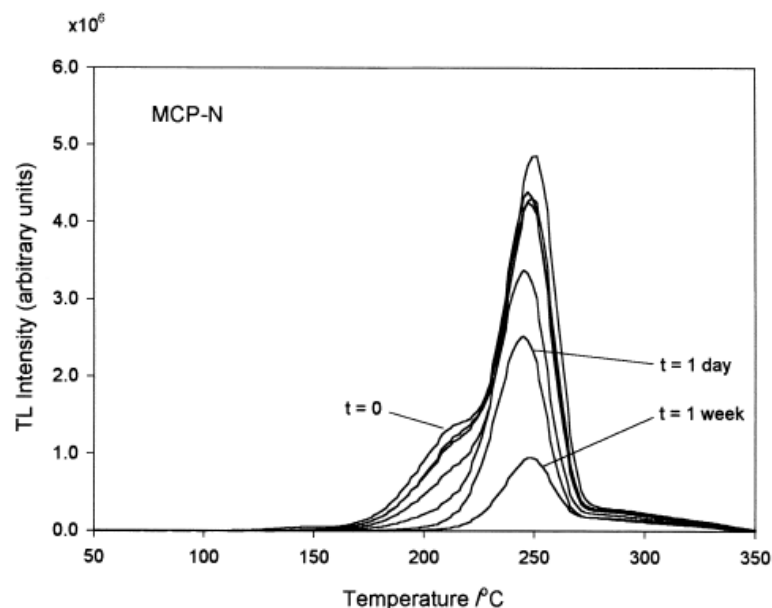


Figure 6

Fig. 2. The effects of light exposure from an arc lamp on LiF:Mg,Cu,P (MCP-N) glow curves. Exposure times are shown in Table 2.

The sensitivity of MCP-N detectors to light were investigated by L.Duggan, M. Budzanowski and others [Dug2000]. The MCP-N detectors were irradiated with 10 mGy  $^{137}\text{Cs}$   $\gamma$ -rays and exposed 2 weeks at ambient light (direct sunlight without any glass protection). The other group of irradiated MCP-N detectors was exposed for the arc lamps (Rofin system with a 200 W Hg arc lamp) for the time from 5 min to 2 weeks.

Approximately 50% loss of signal for MCP-N detectors was observed after 2 weeks exposure on the direct sunlight (summer-time, Poland). Approximately half the thermoluminescence signal was lost after 1 day exposure under the arc lamp.  $\text{LiF:Mg,Ti}$  (GR-100) and  $\text{LiF:Mg,Cu,P}$  (GR-200) detectors were found more sensitive to light and were losing 50% of TL signal after 5 hour arc lamp exposure.

### 8) Sensitivity to other environmental conditions

The specification for dosimetric cards of the HPA service [HPA2005] requires that response should not change by more than 20% after 48 hours storage at temperature ranging from  $-10^{\circ}\text{C}$  to  $+40^{\circ}\text{C}$  and relative humidity ranging from 40% to 90%. The influence of environmental conditions on the response of TLD-EC cards was tested in the following experiment. Nine cards have been prepared by annealing in a reader using standard TTP and exposed to a dose of 1 mSv. Then, three of them were stored in an oven at  $+40^{\circ}\text{C}$ , next three were stored in a freezer at temperature of  $-20^{\circ}\text{C}$  and last three were stored at room temperature. After 48 hours all cards were read out. The results are presented in Table 3.

Stability of MCP response in temperatures ranging from  $-20^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$  was also studied by Saez-Vergara *et al* [Sae1999]. They found an excellent stability of the main peak (within a few percent) at all temperatures over three months period.

Table 3.

Relative response of TLD-EC cards stored 48 hours at different temperatures, with respect to room temperature.

Temperature [ $^{\circ}\text{C}$ ]	Position 2	Position 3
+20	1	1
+40	0.995	0.998
-20	0.982	0.954

Influence of humidity on the response was not tested in a systematic manner. However, the twenty years long experience with using MCP detectors in laboratories of the Institute of Nuclear Physics, during which period various climatic conditions were present, enables us to draw conclusion that humidity has negligible influence on these detectors. This conclusion is further supported by lack of any reports or remarks on such effects from any of users of MCP-7 detectors, which are in use on all continents.

According IEC1066, the stability of dosimeter under various climatic conditions should be tested after storage (i) under standard test conditions (30 and 90 days), (ii) storage at  $50^{\circ}\text{C}$  and 65% relative humidity (30 days) and (iii) storage at  $20^{\circ}\text{C}$ , 90% humidity (30 days).

## 9) Stability

One of the important properties of thermoluminescent detectors, from the point of view of routine applications, is stability of their characteristics during several re-uses. For LiF:Mg,Cu,P detectors it was frequently reported in the past that they may show a loss of sensitivity over repeated application, what was regarded as the main disadvantage of these detectors. To test the performance of the TLD-EC cards, ten pieces samples underwent 46 cycles of irradiation with 1 mSv dose and readout. The obtained results are presented in Figure 7. To minimize influence of changes of reader sensitivity, the response of TLDs was normalized to the readout of the reference light source, which was performed at the beginning and at the end of each measurement cycle (however it seems that this still not compensated all fluctuations of the reader performance). After each ten cycles an additional readout without irradiation was performed to remove any residual signal.

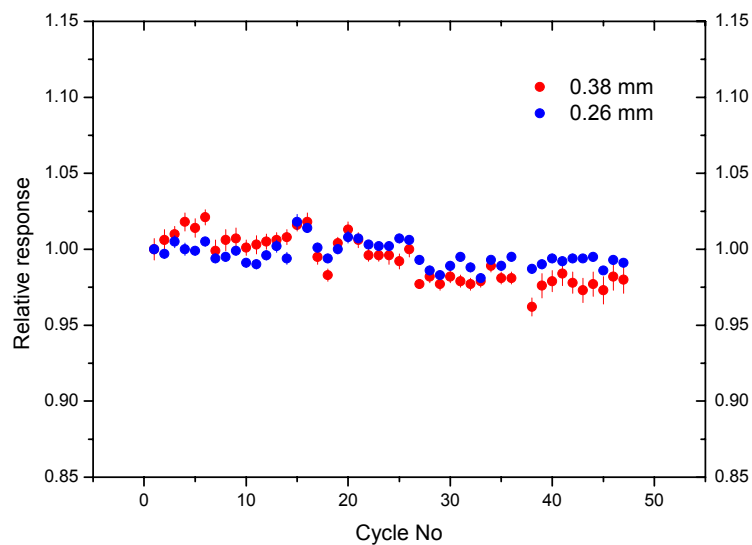


Figure 7. Response of TLD-EC cards over repeated re-uses, with respect to the first measurement

The only observed decrease of the response is an abrupt change which occurred at cycle no 27. This seems to be a reader effect, because before and after this cycle the response is quite stable. Anyway, the conclusion is that the tested cards show no trend of sensitivity loss. Similar results were obtained also in the previous study [Bud2005].

## SUMMARY AND CONCLUSIONS

Selected dosimetric properties of TLD European Card, which are of relevance for personal dosimetry, were investigated or evaluated on the basis of results of the previous research by the accredited Laboratory for Personal and Environmental Dosimetry at the Institute of Nuclear Physics, Krakow, Poland. The cards, compatible with Harshaw/Bicron TLD card were evaluated on the Harshaw reader Model 8800. The cards were equipped with 2 high sensitive MCP-7 (LiF:Mg,Cu,P) detectors (0.38 mm thick in position 2 and 0.26 mm thick in position 3).

Detection threshold was found on the level of 0.97  $\mu\text{Sv}$  (0.38 mm) and 1.68  $\mu\text{Sv}$  (0.26 mm), which is approximately 2 orders of magnitude less than the 100  $\mu\text{Sv}$ , given by IEC 1066. Dose sensitivity was found to depend on the TLD reader settings and varied from 10.3 nC/mSv (0.38 mm) and 6.87 nC/mSv (0.26 mm) for 500 V at PM tube to 338 nC/mSv (0.38 mm) and 224 nC/mSv (0.26 mm) for 800 V at PM tube. The linearity response in the dose range between 0.1 mSv and 2800 mSv was within  $1.00 \pm 0.05$ , which fulfills the ICE requirements with margin of 0.05. The residue dose was found to be about 1% for 0.38 mm and about 0.8% for 0.26 mm detectors. The residue after 0.7 mSv was 9.3  $\mu\text{Sv}$  (including background noise). The fading was found 5% per year at the temperature 33°, and is lower for room temperatures. The sensitivity for thermal neutrons was found  $1.3 \cdot 10^{-9}$  mGy/(neutr/cm<sup>2</sup>), which was the lowest value for all investigated lithium fluoride detectors. The effect of environmental light on zero point i.e. the increase of TLD signal background signal, observed in the non-irradiated detectors, after indoor exposure were found between 9 and 12  $\mu\text{Sv}$  for 24 hours exposure. Influence of environmental conditions: storage at temperatures +40° C for 48 hours lead to decrease of TL signal 0.2 to 0.5%, in -20 °C up to 4.5%. Stability: after 47 readouts, the change of TL signal was not greater than then 2.5%. Part of this effect could be attributed to the TLD reader instability.

The investigations of TLD –EC dosimetric cards with MCP-7 detectors proved their superior properties for personal dosimetry. Replacing the standard LiF:Mg,Ti material into the high sensitive LiF:Mg,Cu,P allowed to reach much lower detection threshold, improved photon energy response, decreased sensitivity to neutrons and enhanced the range of linearity.

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