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Injection molding scintillator for ATLAS Tile calorimeter

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Abstract

Current status of injection molding technique as well as scintillation tiles measurements are presented.

1 Introduction

The construction of modern very big experimental setups in high energy physics requires large amounts of plastic scintillator. Standard technology for scintillators production use styrol polymerization inbetween glasses. Complicated procedures for glass surfaces preparation, long polymerization time lead to high price and long production time of such kind scintillators.
On the contrary, injection molding technique is well adopted for mass production of moderate size tiles. The production rate is high, the mold price itself doesn't determine final scintillator price being a few times less as compared to the standard commercial one. Somewhat worse light attenuation length do not affect calorimeters energy resolution.

2 Injection molding

Injection molding technique is widely used in industry. Most of the plastic goods, details, packings etc. are produced with it's usage. The application of injection molding technique for mass production of scintillation tiles had been first developed at IHEP, Protvino in early eightyth [1]. Commercially available optically transparent granulated polystyrene (PS, the grain size about 3 mm) is used as a scintillation matrix base. Polystyrene is mixed with finely dispersed scintillation dopants, most commonly used are 1.5% of paraterphenyl (pTP) and 0.04% of $POPOP$ (see Appendix for chemical formula). The choice and concentration of wave length shifting dyes (WLS) depends on the specific requirements (direct or WLS fibers readout, best match of absorption–emission spectra, spectral sensitivity of the photodetector used etc.) and are under consumer's control. The prepared mixture is loaded into molding machine hooper, where it is directed continuously into heated screw cylinder while being mixed. At the exit from the cylinder the temperature reaches about 200^0C and melted polystyrene accumulates in its nozzle. As it becams full, an injection into the mold starts. It lasts about 3 sec at the pressure about 700 atm. After the mold cooling up to about 50^0C, it opens and the tile is taken away. The whole cycle lasts less then 2 mins per tile. Maximum production rate of tiles achieved so

far is 1 min per 250g tile.
The mold can be of arbitrary shape, providing the tiles with grooves and holes if required. Some heat resistant elements (e.g. special WLS fibers) may be put into the mold before injection, resulting in them being embedded inside the tile. In general no additional mechanical treatment (like polishing) is needed for the final product. The tiles dimensions reproducebility is determined mainly by the stability of thermal shrinking. The safe linear tolerance coefficient is about 0.0004. Largest tiles produced so far have dimensions $400 \times 200 \times 3mm^3$. Standard commercial molding machines are being used for the present.
Natural scintillator ageing and its radiation hardness depends on the components used. An important factor is the presence of unpolymerized monomer in a polystyrene. According to the specification of PSM-115 polystyrene (Russian trade mark), it doesn't exeed 0.1%, which is a few times less in comparison with standard bulk polymerized scintillators. Up to 10 years exploitation at IHEP different experimental setups with the usage of molded scintillators do not demonstrate any detorriation of calorimeters performance.
Different factors affect tiles quality and production rate. The quality of the mold's internal surfaces, which form tile's ones, should be mirror like. Not necessary as good as in telescopes, but one should clearly recognize his reflected image. They must withstand contact with melted polystyrene and dyes over long time period. Periodical cleaning of deposits inside the mold during production is recommended. Mold's thermostabilization is necessary for the intensive work at high production rate. There exist different approaches in the injection gate design: tabs or film, parallel or perpendicular to the tile's plane. To our feeling their choice depends mainly on the availables molding machine and mold production technology and on the designer's traditions.
The molding machine must have locking force high enough to keep the product of the injection pressure and opposite tile's square. High stability of the operating regime will assure high reproducebility of the tiles properties.
Wave shifting additivies, pTP and $POPOP$ are easely destroid at high temperatures. At the same time the temperature should be high enough to dissolve WLS dyes and to assure high melt flow. It's important that the very last heater of the screw cylinder should be as close to the nozzle as possible to minimize melt cooling during injection. The process of polystyrene heating and mixing inside cylinder should be minimized because WLS destroing is proportional not only to the temperature but to the time as well. The melt flow and injection gate should assure minimal injection time. But one should remember about additional melt heating when passing narrow gate system of the mold. And it is proportional to the injection's pressure and rate. Reasonable compromise of the above mentioned factors should be found to optimize tiles quality and production rate.

3 Scintillator for calorimeter prototypes

About 5 production batches of scintillation tiles of the total weight about 1t were done at IHEP for Atlas Tilecal prototypes [2]. With the same 3mm thickness of the tiles, two tile's geometries were used. At the begining of prototypes construction all the tiles had the same width 100 mm but 18 different lengths ranging from approximately 200 to 370mm. In addition at the early stage of prototypes tests the

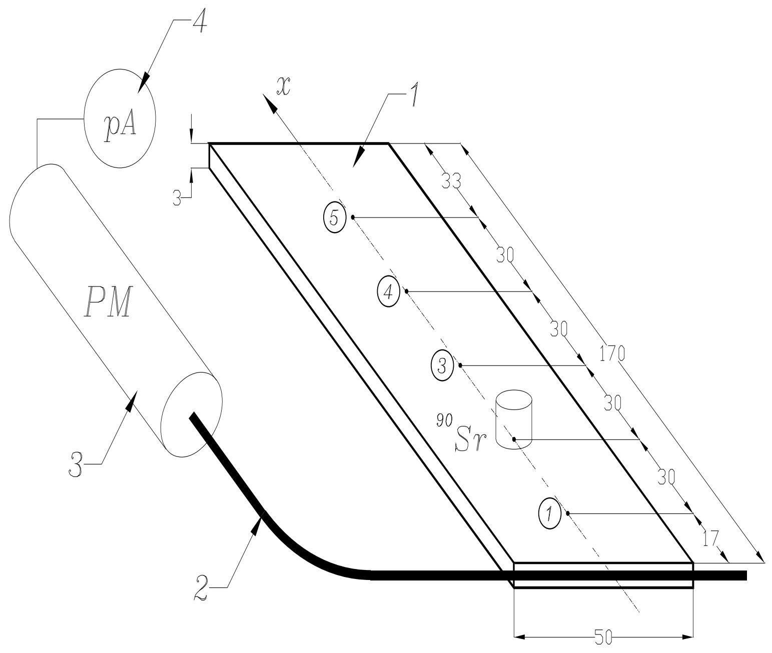

Figure 1: Test setup for tiles measurement.1–tile,2–WLS fiber.

tiles were with grooves for fibers holding and 45^0 chamfers near them. Later, as laboratory tests confirmed, the chamfers were removed and the tile's edges opposite to the fibers became flat. In 1996 the tiles geometry changed, the tile width became not the same but varying from approximately 100 to 200mm with 11 tile's types in total. All the productions were done with the single mold for the first tile's geometry and with another single one for the second. To provide different tile sizes, the molds had exchangable details. All the first geometry tiles were produced with old-fashinate injection molding machine with not very stable operating regime.
During production the quality of the tiles was continuously monitored with the setup illustrated schematically in Fig.1. It consists of a "black box" where the tile was put in contact with WLS fiber. Scintillation light was excited by collimated (4mm in diameter) ^{90}Sr radiactive source, then collected by Kuraray $Y11$ WLS fiber and the current of $FEU-84$ photomultiplier with green extended photocathode was measured. WLS fiber was always the same, periodical measurements of reference tiles were being done to normalize the data. Indeed we have 3 setups of similar design: The setup for the production control by the measurements of unwrapped tiles. Here the source was moved along tile's center by light tighted rod. The setup for the scan of wrapped tiles. Here the tile was in touch with made of black paper upper wall of the box with moved by hand source outside it. In third setup the source movement inside the box was computer controlled.
Some data are presented in Fig.2, where all the tiles of the biggest size were measured

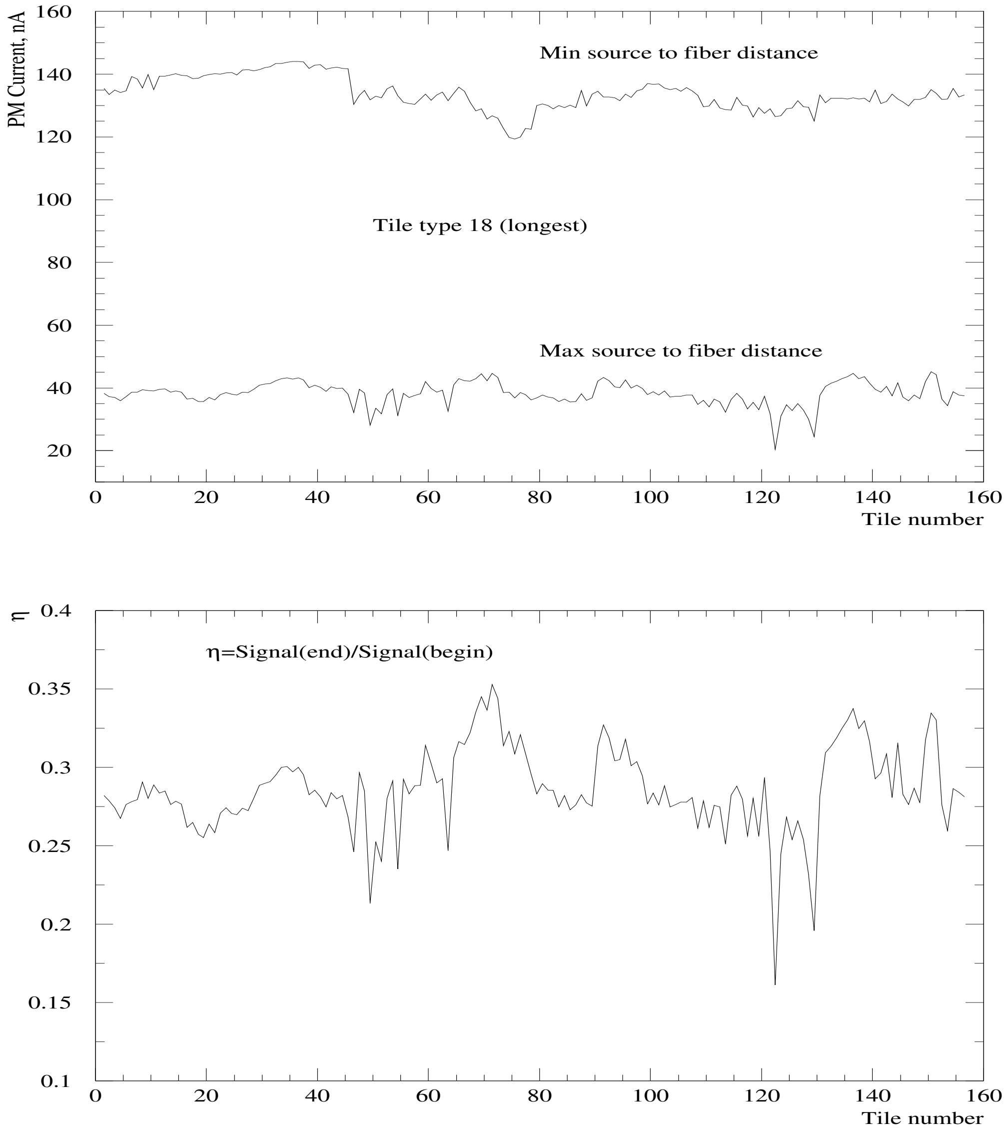

Figure 2: '95 production quality control for the largest tile.

one by one during production process. Radiactive source was put near WLS fiber (light yield) and at the maximum distance from it (light yield and light attenuation length). One can see variations of tiles quality during production caused by the instability of injection regime. Only the tiles with response within about $\pm 10\%$ of the mean value were selected for the usage in calorimeter. Mean values of light yield for all the 18 tile's sizes are depicted in Fig.3.

In 1996 the production of larger tiles of another geometry was done. More modern computer controlled injection molding machine was used. Operation regime of this machine was very stable, periodical control measurements of each 10^{th}–20^{th} tiles didn't demonstrate significant fluctuations in light yield. But the quality of polystyrene used this time was worse as compared to '95 production, see Fig.4. Tile response depends not only on its quality, but on that of wrapping as well. Tiles wrapping refers to inserting them into $Tyvek$ sleeves and installing mylar strips over

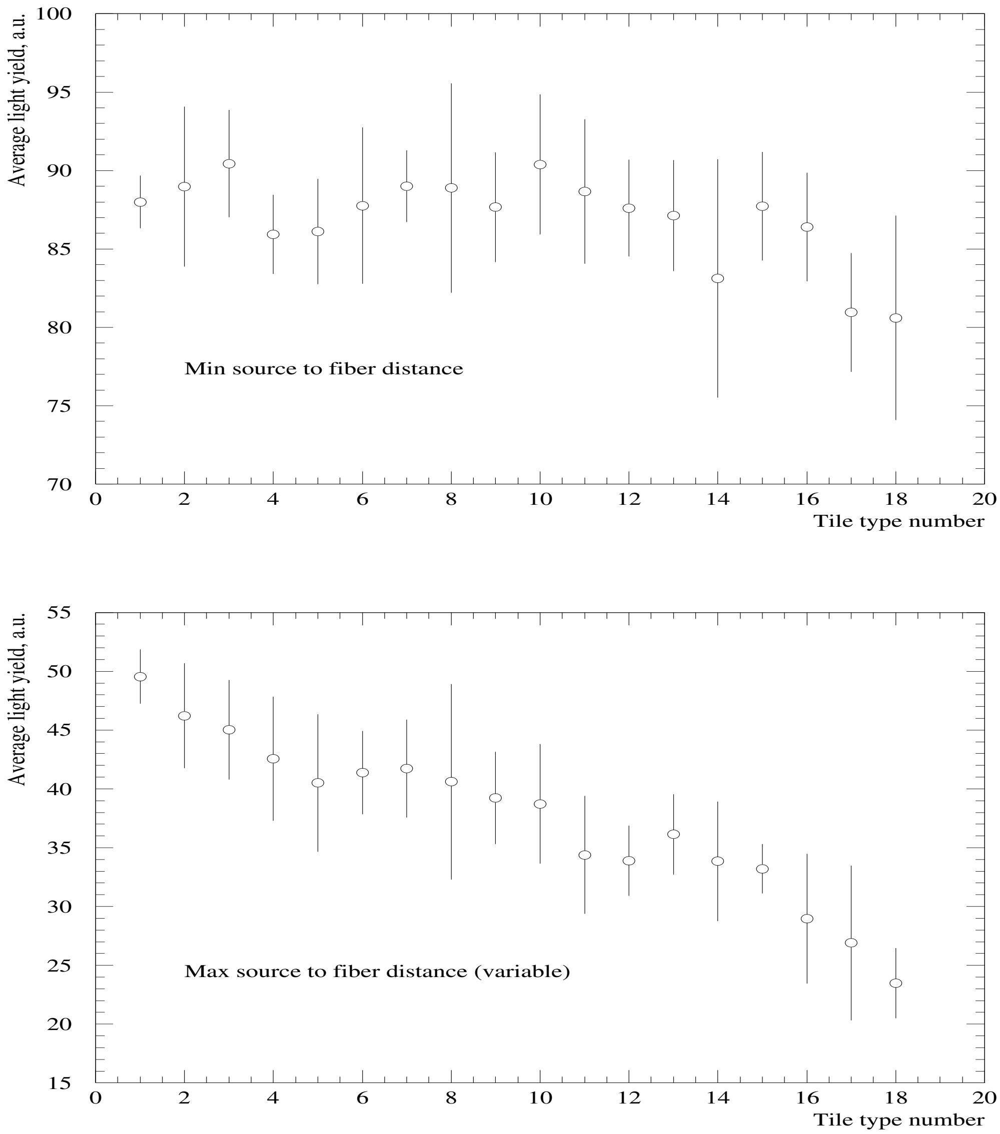

Figure 3: Quality control for '95 tiles production.

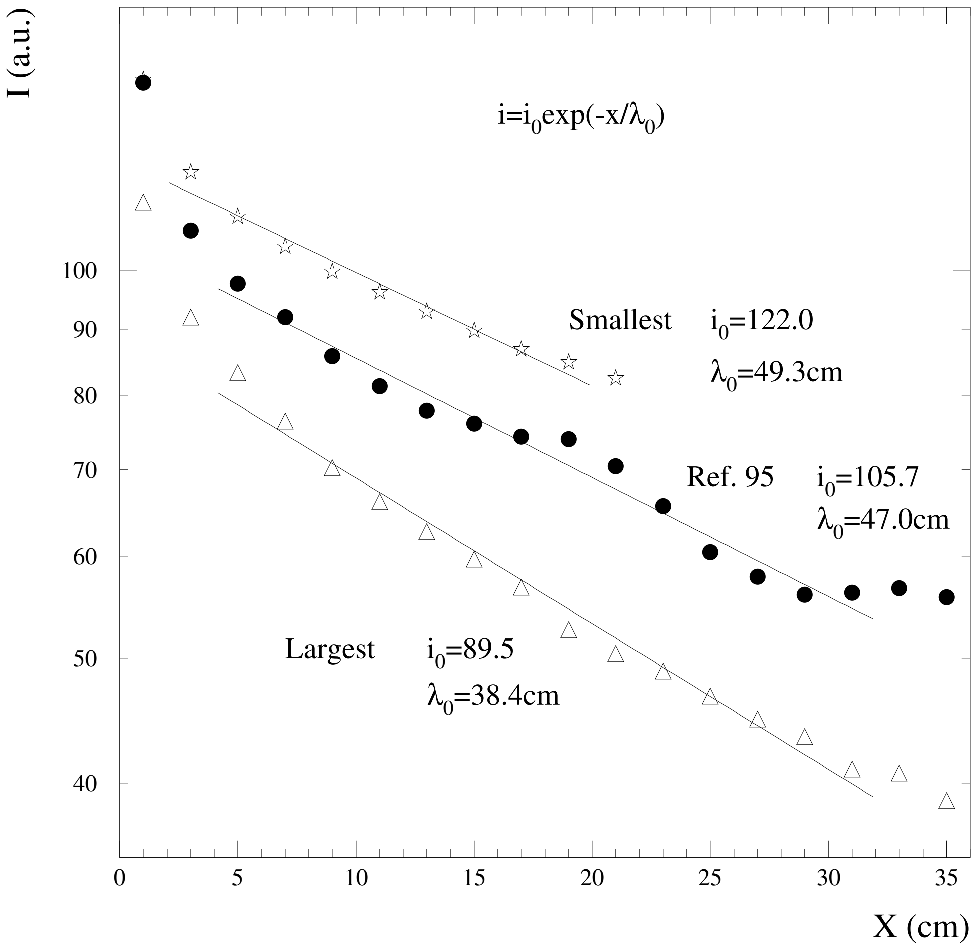

Figure 4: '95 and '96 production comparison.Open points–'96

both ends to couple the tile to WLS fibers. The tiles from size # 6 to size # 11 (the biggest ones) were also "masked", namely the $Tyvek$ sleeves were black-painted where the mylar had to be applied in order to smooth the light yield near the edges. In order to study wrapped tiles uniformity, the light yield from samples of 10 tiles of each of 11 sizes was measured. The results are reported in Figs.5–6 which show that the tiles examined are uniform in their performances at the level of few percents. Another comparison of '95 and '96 tiles wrapped in the same way was done. In

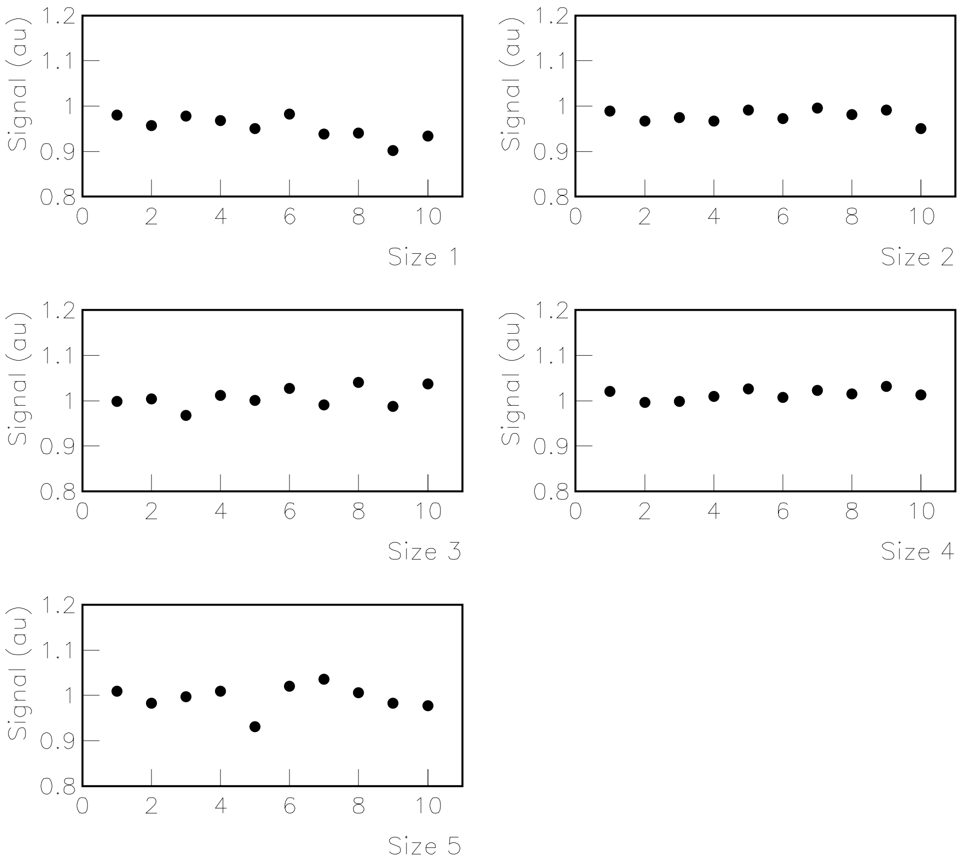

Figure 5: Uniformity of the tile light yield for '96 tiles # 1–5 (unmasked) .

lower part of Fig.7 the summetrized result, as if the tiles were readout by two fibers from both end is shown ($I_{tot}(x) = I(x) + I(l-x)$, where l is the tile's length). One can see slightly worse quality of '96 tiles due to that of polystyrene used.

Apart from the production of standard composition tiles for calorimeter prototypes (PSM-115 + 1.5% pTP + 0.04% $POPOP$), some investigations of alternative base materials and concentrations were done. We observed little dependence of the light yield on changing $POPOP$ concentration from 0.02% to 0.1%. There was

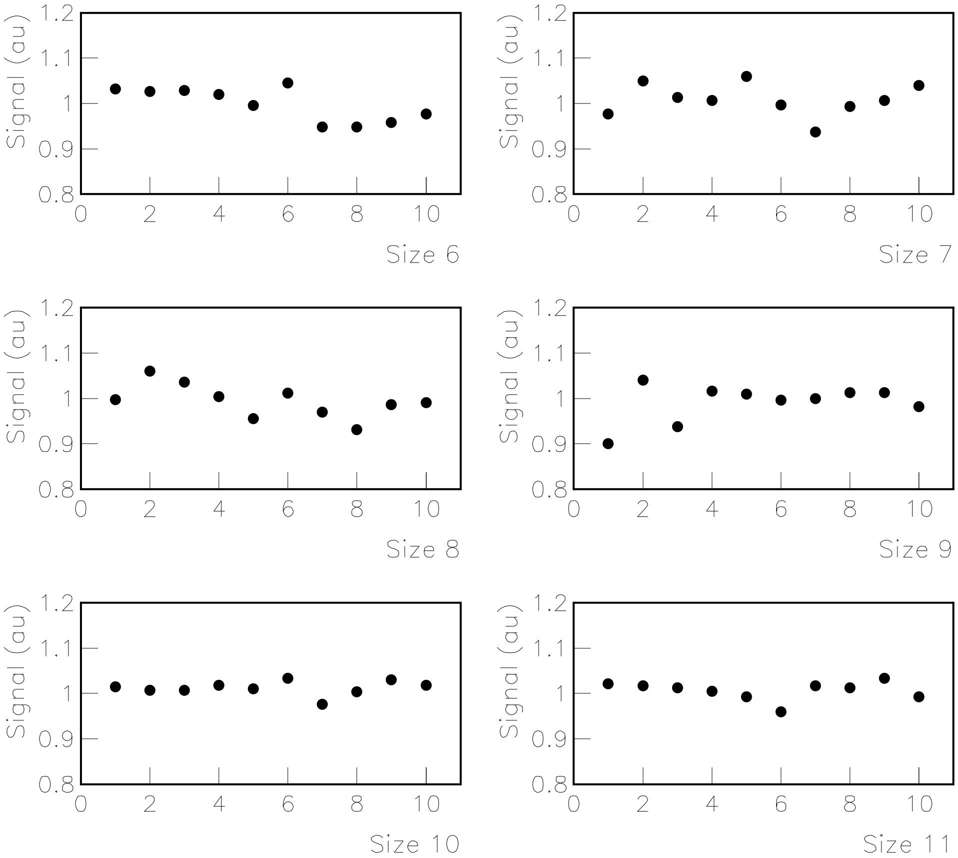

Figure 6: Uniformity of the tile light uield for '96 tiles # 6–11 (masked).

about 10% increase in light yield when increasing pTP concentration to about 2%, but pTP cost is significant fraction of the scintillator one.
Test results of the scintillators with other WLS dyes are presented in Fig.8. One can see that PPO may be a good candidate to replace pTP. We didn't observe significant differences between "western" (FLUKA) and "russian" made wave length shifters of the same type.

Our experience in tiles production clearly demonstrated strong dependence of scintillation tiles transparency on that of polystyrene. We tried to investigate another polystyrenes, some randomly choosen Czesh (Krasten) and British Petrolium (BP) ones. The results are depicted in Fig.9. Up to now Russian made PSM-115 polystyrene seems has the best transparency. According to the specification another polystyrene PSM-151 has better transparency (though the exact meaning of transparency isn't clear). The transparency of polystyrene can be measured with the spectrophotometer. One may prepare the solution (up to 20% by weight

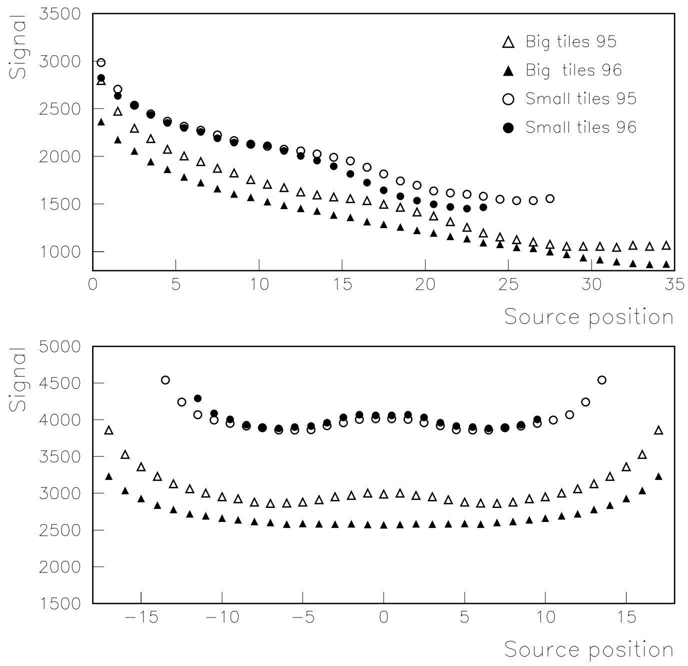

Figure 7: '95 and '96 tiles comparison.

concentration) of polystyrene grains in e.g. *tolyene* which has refraction coefficient close to that of polystyrene. Or produce pure polystyrene tiles without adding WLS dyes. Some results are shown in Fig.10. The comparison of two PSM-115 samples from different production batches with PSM-151 one show transparency drop near the maximum of pTP emission spectrum. The attenuation length of pTP's light is small, some millimeter. This light should be reemitted by $POPOP$ either directly or after reflection from the wrapping material. Note that the wrapping is only essential for the being reemitted light. Blue light emitted by POPOP and escaped from the tile will never be trapped back. One can cure this transparency drop by increasing pTP concentration, but it will increase scintillator cost. In addition PSM-151 has four times less melt flow index, which may complicate injection process. PSM-115 is general purpose polystyrene, not an optical one. And so there is some spread in its transparency at hard blue wavelengths for different production batches. Most of the product in the market is usually produced surface treated with some wax, which obviously spoil its natural transparency. Another constrain is that polystyrene should be "fresh". There are indications that after long term storage it looses the quality. The search for stable supplier of transparent polystyrene is an important goal for large size tiles production.

According to Ref.[3], the reflectivity of different types of $Tyvek$ fiber's paper differs greately. Reflectivity of $Tyvek\ L-1085B$ is almost two times better as compared to the material we used. Though it is 2.5 times thicker, which for the fixed absorber's

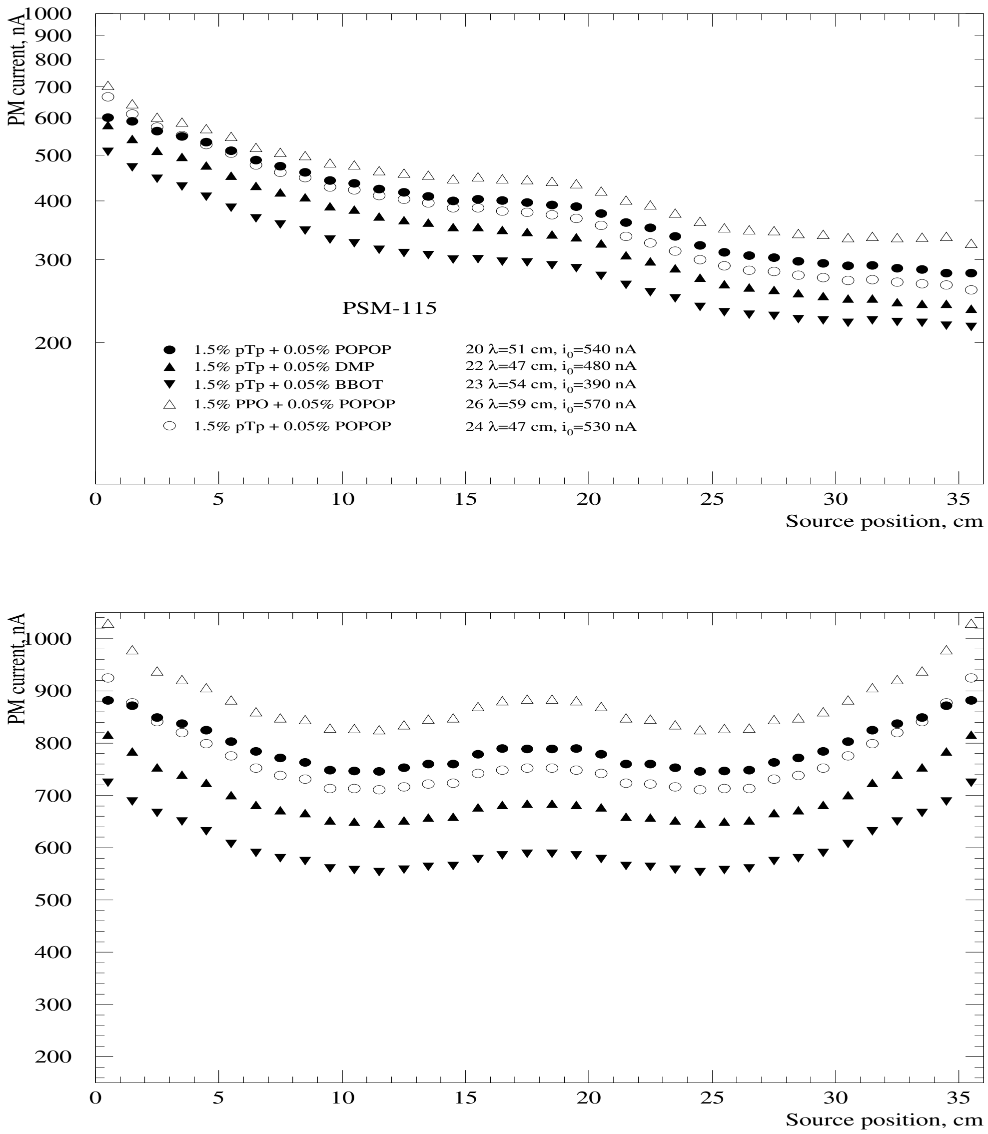

Figure 8: Tiles light yield for different WLS dyes. 1 and 2 fibers readout.

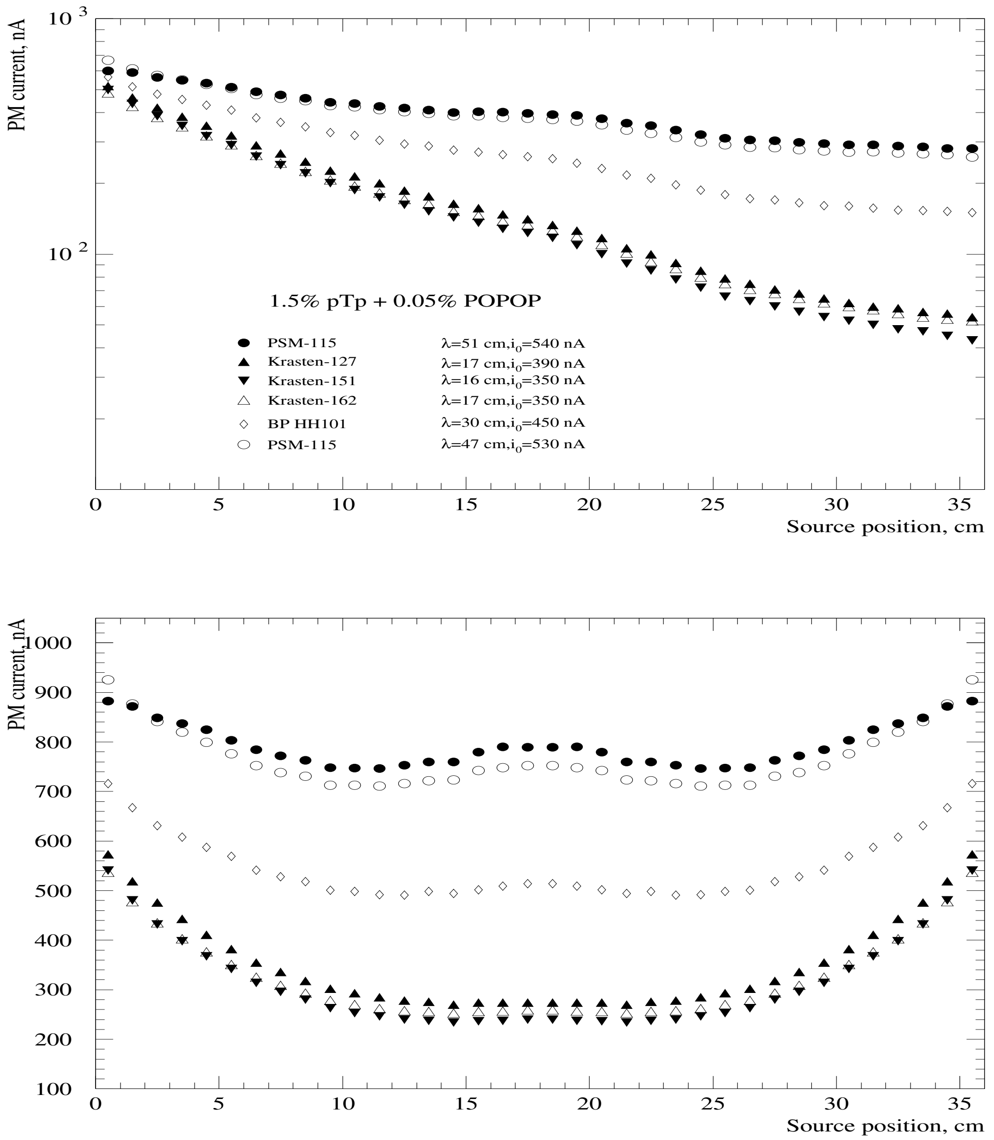

Figure 9: Tiles light yield for different polystyrenes. 1 and 2 fibers readout.

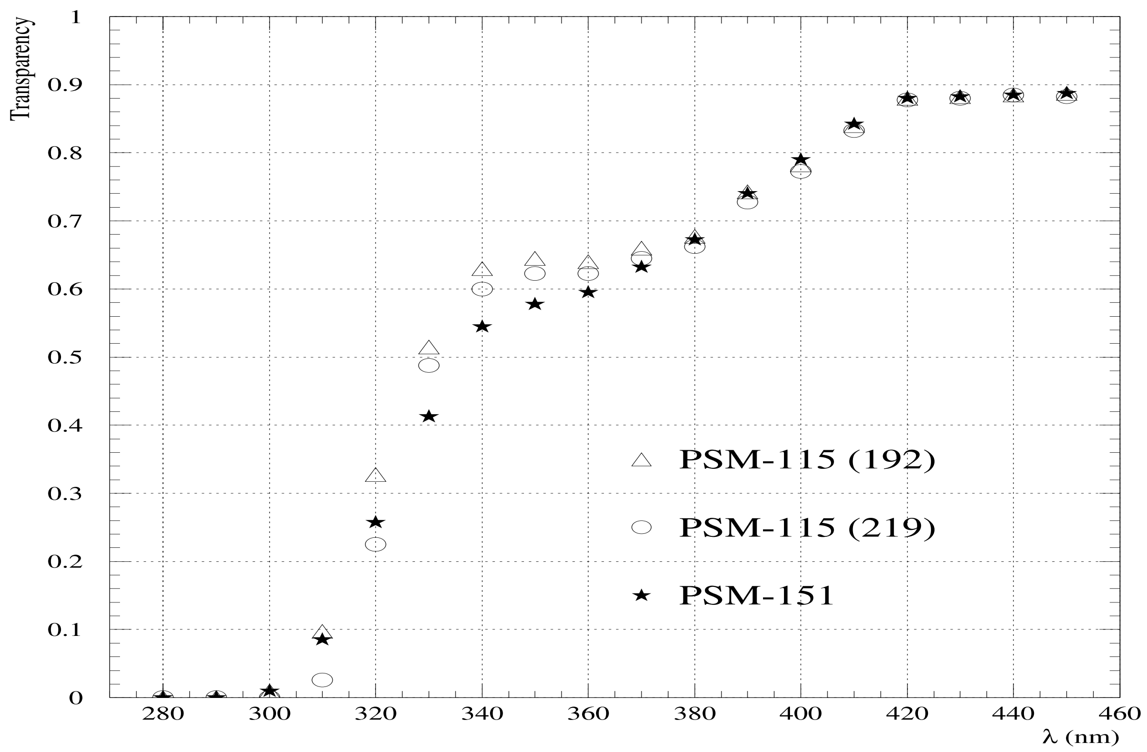

Figure 10: Pure polystyrene tiles transparency.

gap width of the calorimeter will require thinner scinntillation tiles, the final light yield may happen to be in favour of this material usage.

The ATLAS setup is planned to be operated during about 10 years. By this reason it is necessary to evaluate the deterioration of the properties of some setup's components, in particular scintillation tiles for that time. There exists a way to estimate natural ageing of tiles by means of increased temperature action for the acceleration of chemical reactions.

The rate of chemical reaction (i.e. ageing) is determined by many factors, namely by the concentration of reagents, the temperature, geometrical sizes of reagent particles etc. If to consider natural ageing of scintillation tiles as a simple chemical reaction like $A \rightarrow X$ resulting in the deterioration of main quality indexes of scintillator (light yield and attenuation length), then we can write $-d[A]/dt = k(T)[A]$, where $[A]$ is the concentration of reagent A and $k(T)$ is the temperature dependent value known as reaction rate constant. According to Arrenius formula coefficient k is defined as $k = k_0\ exp(E/RT)$, where T, R, E are temperature, gas constant and activation energy correspondingly [4]. Using this formula it is easy to calculate the quotient of chemical reaction rate constants at different temperatures. For instance at room temperature $T_1 = 20^0C$ (natural ageing) and at higher one $T_2 = 70^0C$ (artificial ageing) and with an activation energy for polystyrene E=91.19 kJ/mol [5] this quotient is equal to 235. Using this value and measuring scintillation tile properties after heating one can evaluate possible degradation of scintillator resulting from natural ageing.

The measurements were done in a way similar to that of Fig.1 with the usage of modified graph plotter. The collimated radiactive source mounted on a carriage movable in two directions by plotter frame was able to scan the region of $36.5\times28\text{cm}^2$ with an accuracy of 0.3mm in both coordinates. The photocurrent was measured and digitized with precise multimeter V7-49. Multimeter operation mode control and data readout were performed by means of *GPIB* interface in *SUMMA* standard (*CAMAC* protocol). The *SUMMA* crate contains also customized plotter driver. All operations were performed under control of *IBM PC*. The program had user-friendly window interface allowing to specify the hardware configuration, the coordinate table for measuring points, pause between movements and current measurement conditions.

We kept two samples of molded scintillation tiles wrapped with *Tyvek* at temperature of 70^0C during 25 days with small time breaks for measurements. In accordance with our calculations for the activation energy mentioned above that corresponds to the total natural ageing up to 16 years. Some results are shown in Figs.11-12. Fig.11 presents typical scan lengthwise the center line of a tile with 2 cm pitch. The signal increase in the region of fiber location is not understood yet and probably is caused by the local improvement of surface quality at the tile edge. In Fig.12a–b the relative attenuation length behaviour in time is shown. The stepped rise of attenuation length just after starting of heating may be caused by some annealing processes in scintillation tiles at higher temperature. So taking the first point as a reference one, 5% degradation of attenuation length in the point corresponding to 10 years is observed. Unfortunately the recount coefficient day→year depends strongly on the value of activation energy (as index of exponential function). Fig.13 gives the dependence of simulated tile "age" on activation energy deviation from the value we used. As an example, ±10% deviation for activation energy corresponds to the interval

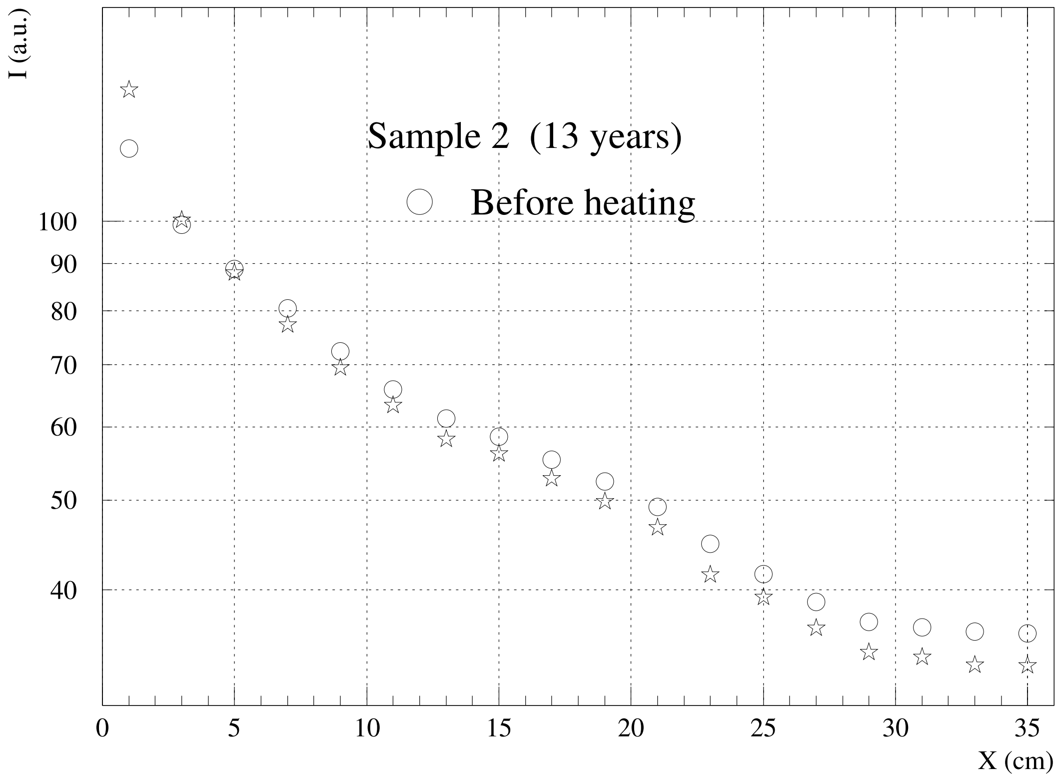

Figure 11: Typical scan of scintillation tile.

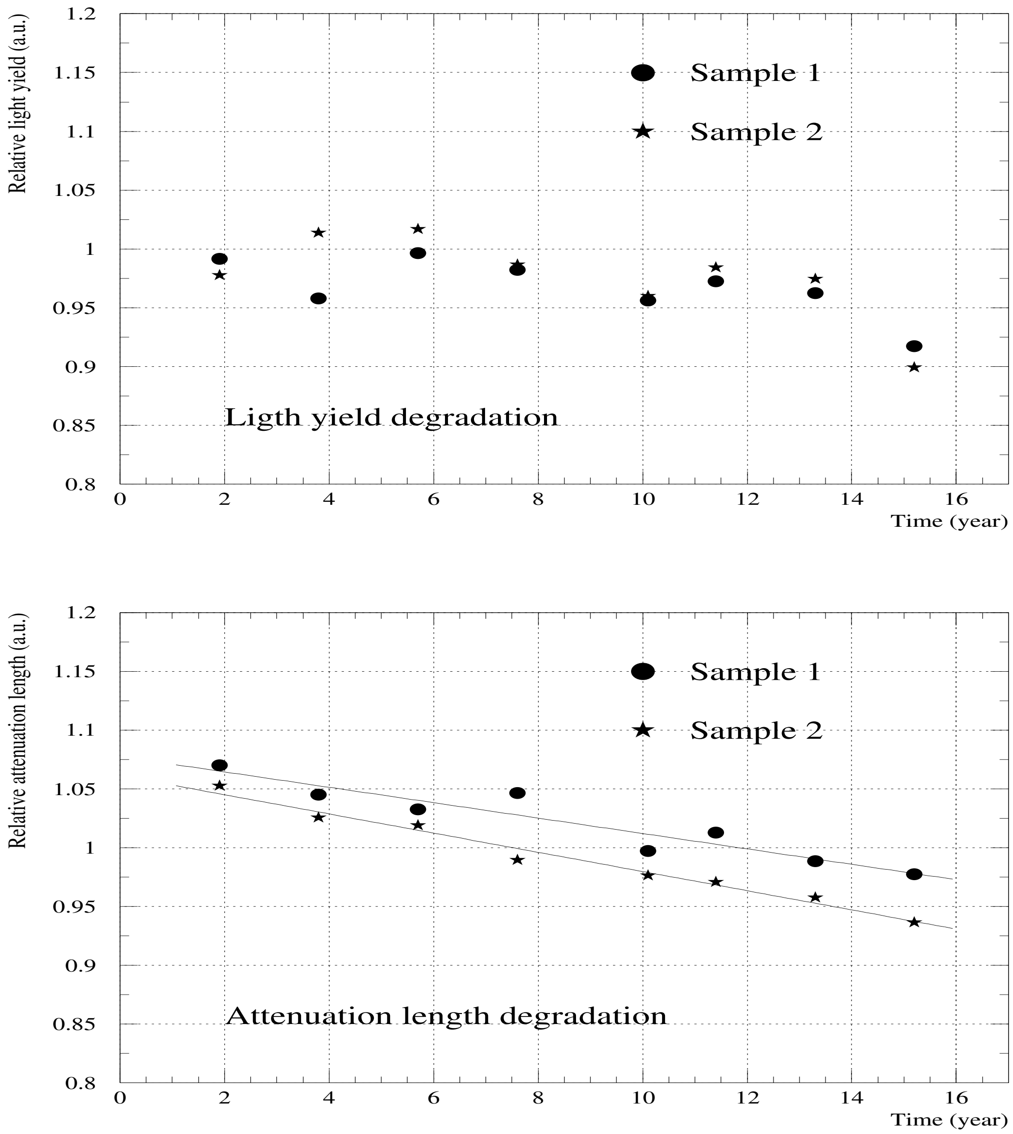

Figure 12: Degradation of scintillation tiles properties.

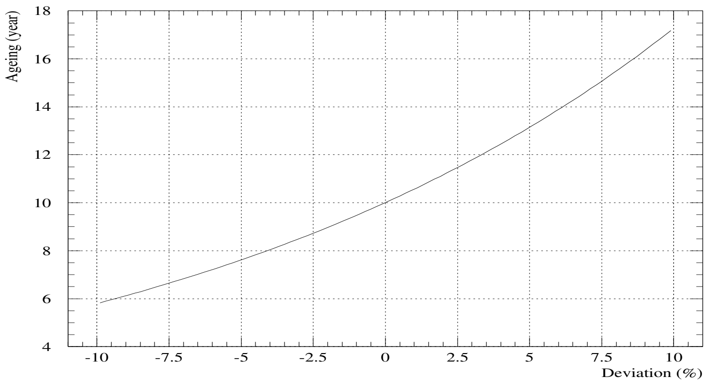

Figure 13: Influence of the activation energy value on the ageing duration.

from -40% to +80% of ageing time. The value of activation energy we used was measured for scintillator with the same fluorescent additives but produced by means of bulk polymerization [5]. To measure it independently the measurements should be repeated at other temperatures. According to our current measurements the total deterioration of scintillation tiles properties caused by natural ageing should not exceed 10% level after 10 years of operation.
Another factor which affects tile's ageing is the surface worsening (e.g. cracks like frost traces on windows). And the heating may smooth down this effect. We have injection molding tiles produced more then 10 years ago. Their visual inspection didn't find surface defects.
There was the statement that the low temperature cause damage of bulk polymerized scintillation tiles surface. We exposed for a week few injection molded scintillators at the temperature about -20^{0}C during russian winter. No changes in tiles responce were observed after the execution.

4 Radiation hardness

Plastic scintillator modifies its scintillation properties and transparency under irradiation. This modification depends on the type of scintillator, on the way it was produced by and have rather complicated dynamic behaviour due to the processes of destruction and restoration (annealing) of its chemical components. The

molded scintillation tiles based on PSM-115 polystyrene showed the highest level of radiation hardness as compared to PS or *polyvinyltoluene* samples produced by the bulk-polymerization [6]. We used in our analysis scintillation tiles produced by the injection molding technique at IHEP with standard WLS dyes concentration of 1.5% of pTP and 0.04% of $POPOP$. Large size tiles were cut mechanically to dimensions of $3\times50\times170\mathrm{mm}^3$ to fit the volume available for irradiation and were covered with $Tyvek$ paper. The samples were irradiated uniformely in a flux of γ-quanta from ^{137}Cs radiactive sources at a dose rate of 60 mGy/s (1.9 MGy/yr) at room temperature in air. The tiles were exposed at different doses, from 0.65 kGy (3 hours of irradiation) to 10.4 kGy (2 days irradiation). Note that the dose rate in these irradiations was much higher than the one expected in Atlas (0.4 kGy in worst place at maximum luminocity after 10 years). Before and after irradiation the light yield for each tile was measured with the setup of Fig.1. The measurements were done at different distances from the readout fiber (not being irradiated) at regular time intervals starting just after the end of irradiation. For the crosscheck, few tiles were exposed and measured for all accumulated doses. The reproducebility of the results was continuously monitored by few reference tiles measurements. An estimated measurements accuracy was about 1%.
Some of raw data results are presented in Fig.14a–c. One can see quick tiles recovery just after irradiation followed by some degradation and finally stabilization. The dependence of the light yield just after irradiation differs from that after some recovery time. Under real the conditions of calorimeter operation scintillation tiles will be exposed continuously with some breaks between irradiations. We tried to simulate these conditions providing the given dose in one and two steps with a few days break for double irradiation. The results presented in Fig.15a–b show a negligable difference between one and two-step irradiation. One can see that the recovery (annealing) rate is high, some hours. It may justify usage of high irradiation rate for future calorimeters radiation study. The detailed behaviour of recovery process somewhat depends on the materials sample used and injection regime. But the final radiation damage is less affected.
The presence of intensive high energy secondary particles irradiation will be a characteristic feature of many experimental setups for future particle accelerators. The radiation hardness investigation of plastic materials in hadron fluxes is also of great importance. In our case we dealt with secondary particles (mostly soft hadrons) originating from the internal Al-target of 70 GeV IHEP proton accelerator. PS based scintillator samples were exposed in a flux of secondary particles emitted at 30^0 to the beam axis at an average dose rate of about 20–30mGy/s. Doses of γ-quanta and hadron irradiation were measured with polymer-alanine dosimetry [7]. This method provides an experimental accuracy of about few per cent for relative dose measurements while systematic errors of the dose determination were about $\pm10\%$ for γ-quanta and $\pm30\%$ for hadron irradiation. A comparative study of the influence of γ-quanta and hadron irradiation on scintillation tile samples is shown in Figs.16a–b. The recovery processes for the same PS samples are shown in Figs.17a–c. The experimental data indicate that the hadron irradiation may causes higher level of the radiation damage in comparison with γ-quanta irradiation for doses over 1 kGy but it is necessary to improve the accuracy of dose determination for more exact conclusions. Note that the observed difference of radiation properties between these two types of irradiations may allow one to reveal the effect

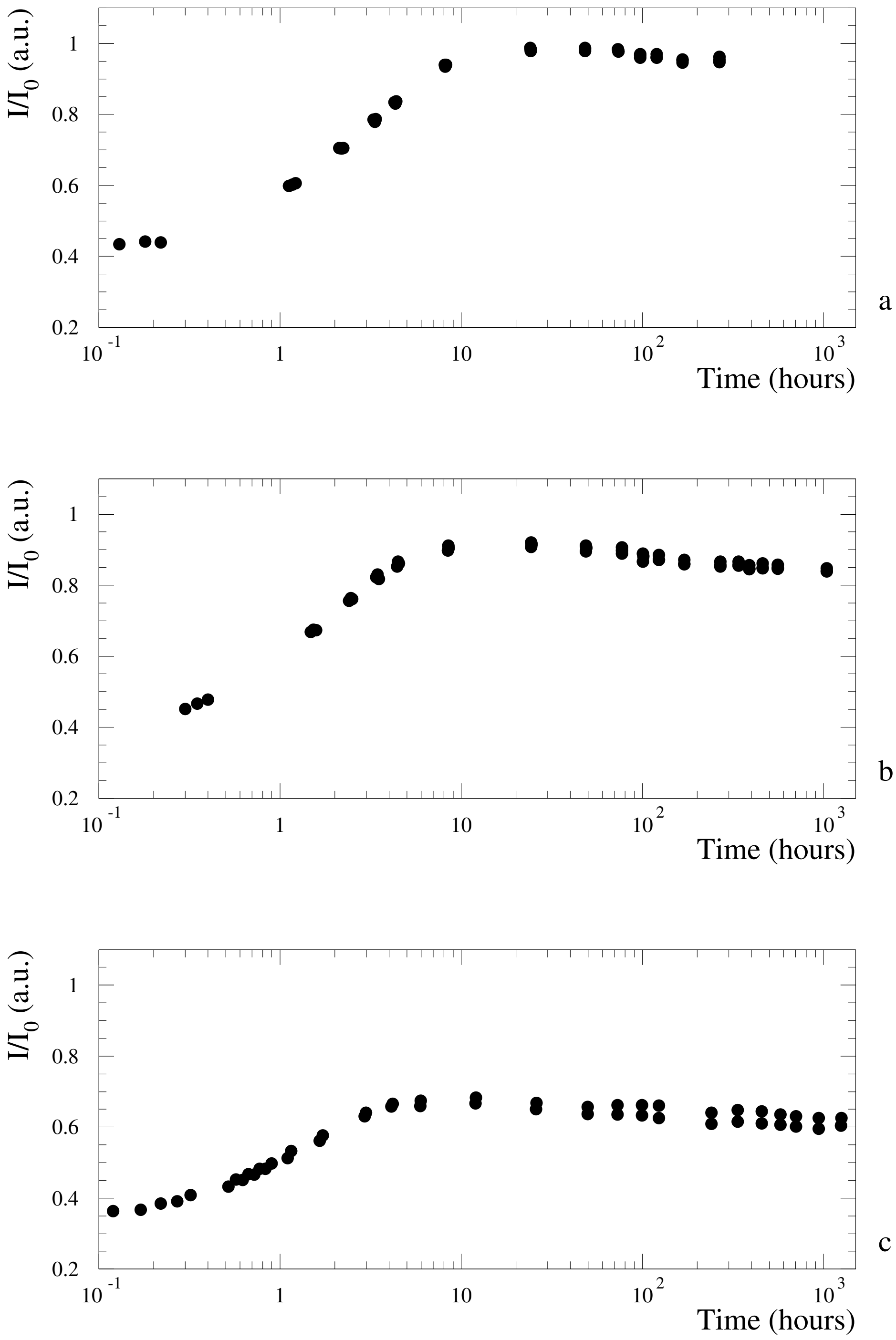

Figure 14: Relative light yield (after/before irradiation) at x=13.5cm versus recovery time for different doses (a–0.65kGy, b–2.6kGy, c–10.37kGy)

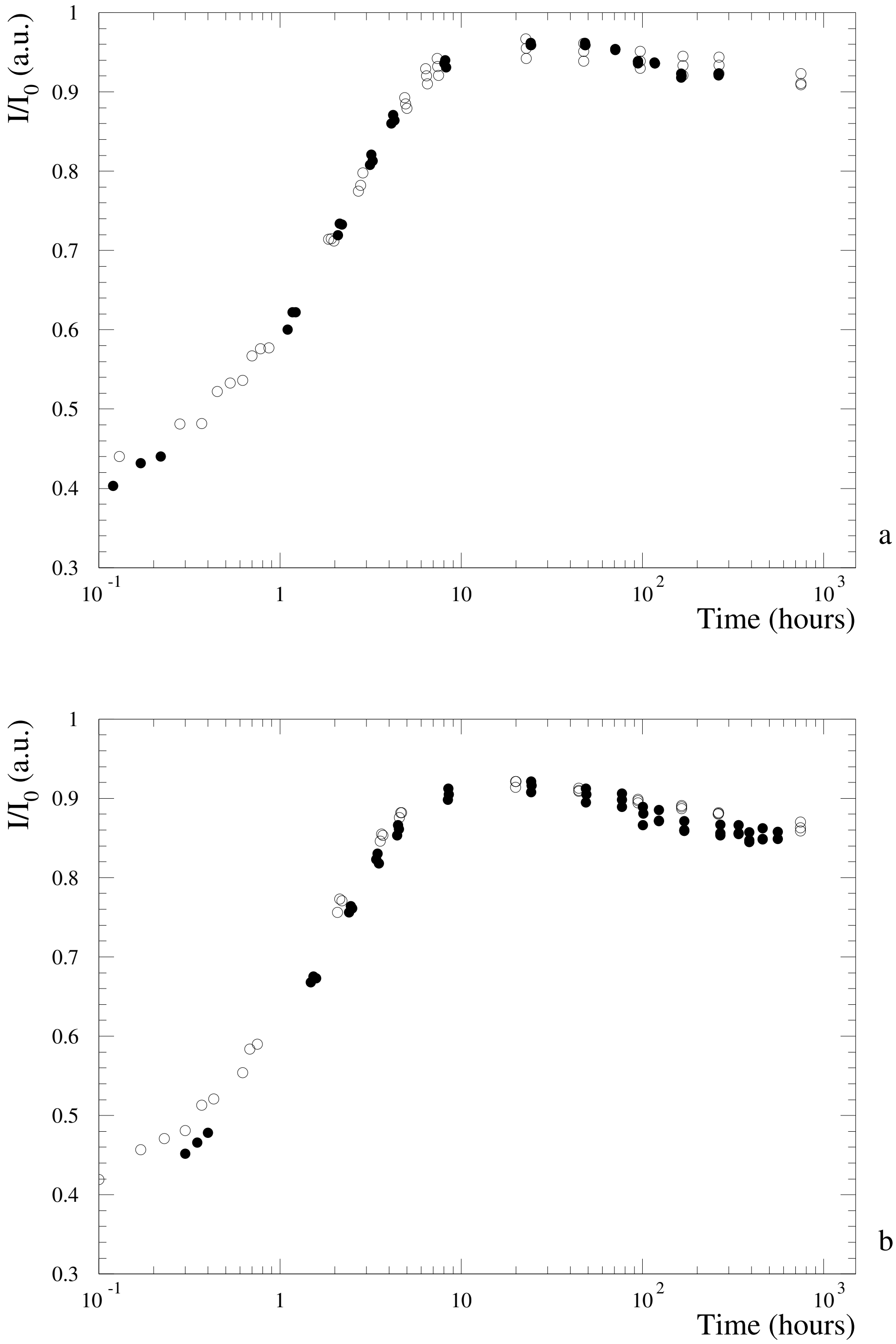

Figure 15: Relative light yield (after/before irradiation) at x=13.5cm versus recovery time for different doses (a–1.3kGy, b–2.6kGy) accumulated in one step (•) and two steps (○)

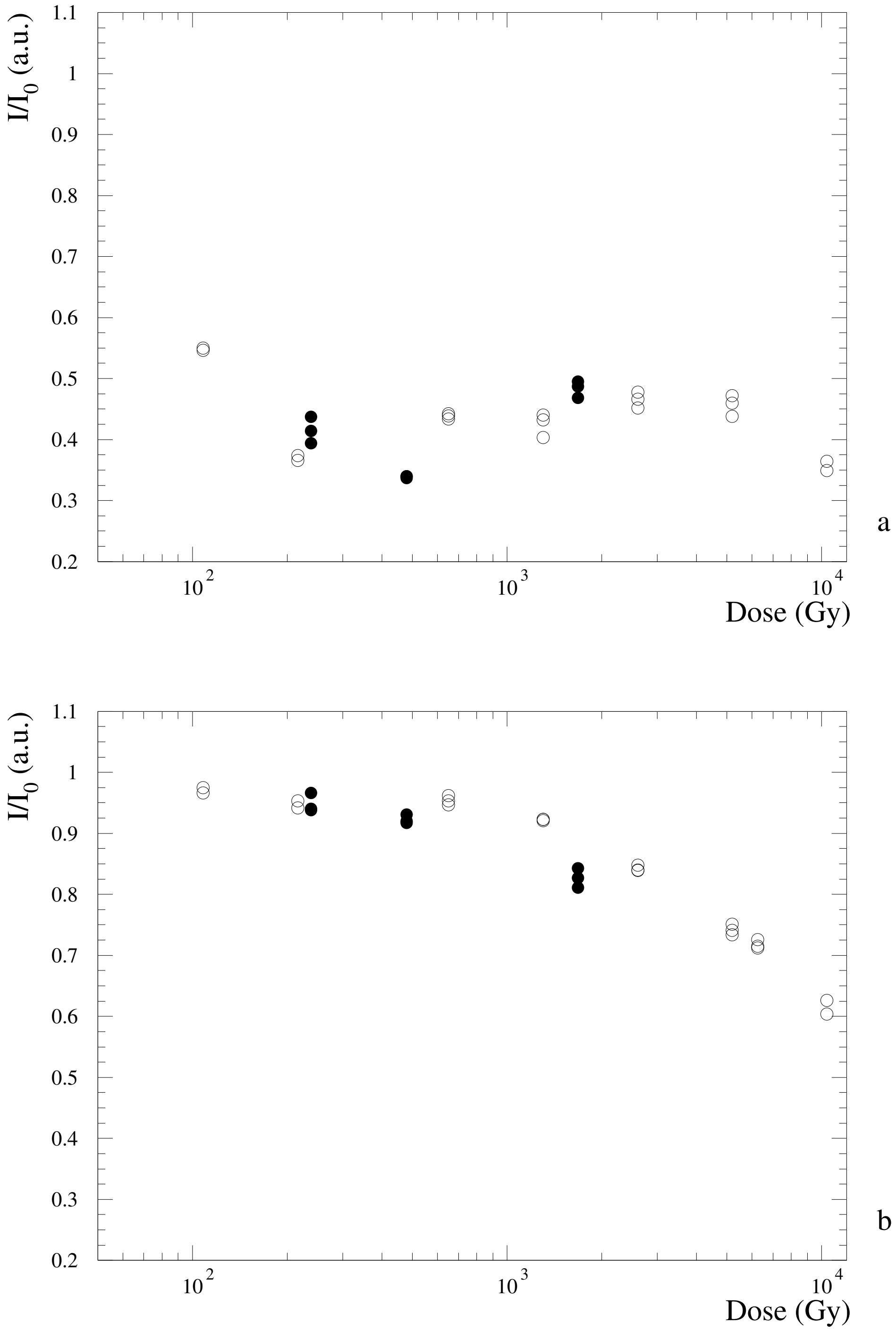

Figure 16: Relative light yield for γ-quanta ($\circ$) and hadron ($\bullet$) irradiation at x=13.7cm versus the dose (a–immediately and b–in a month after irradiation)

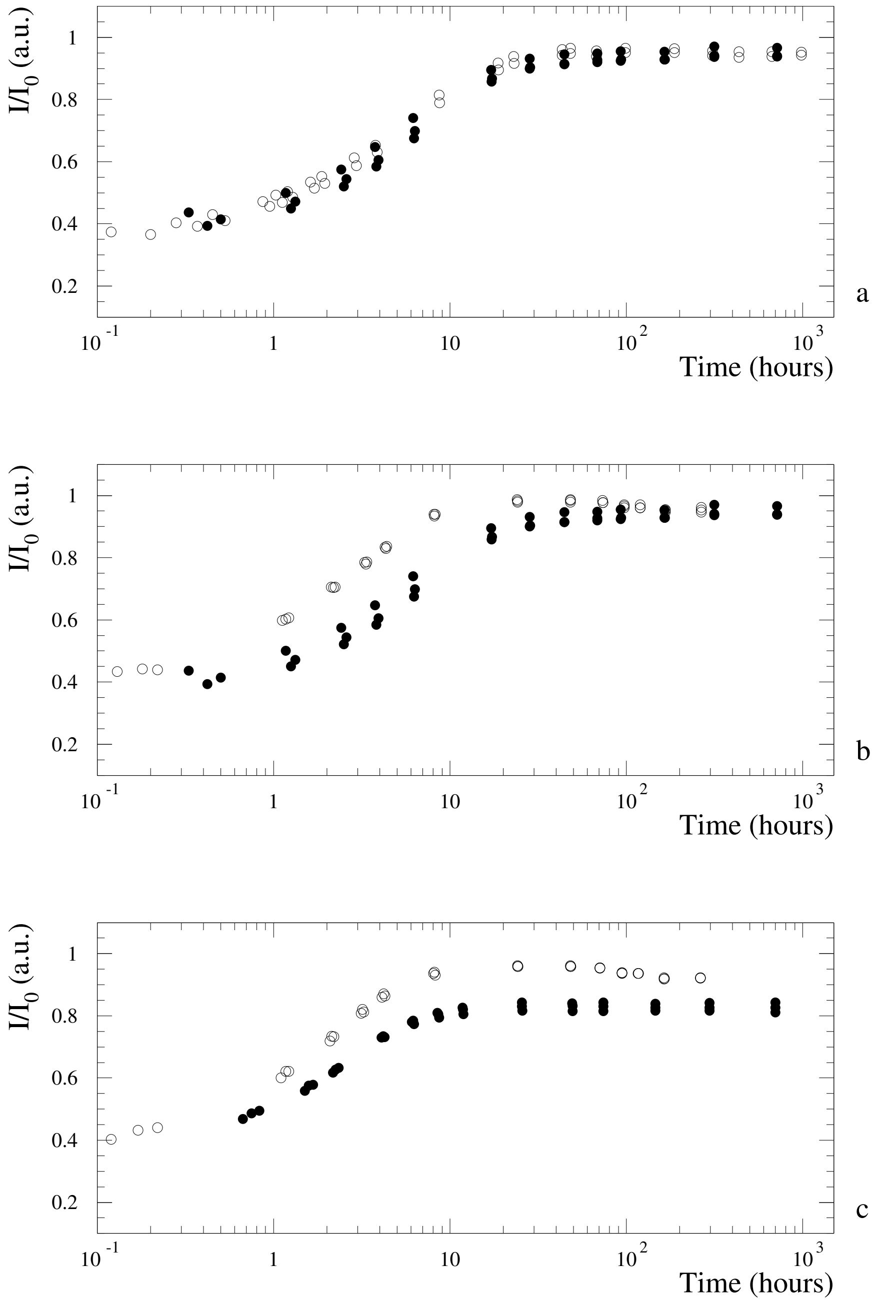

Figure 17: Relative light yield for γ-quanta ($\circ$) and hadron ($\bullet$) irradiation at x=13.7cm versus recovery time (a–after $0.22kGy$ of γ-quanta and $0.24kGy$ of hadron irradiation, b–after $0.65kGy$ of γ-quanta and $0.48kGy$ of hadron irradiation, c–after $1.3kGy$ of γ-quanta and $1.68kGy$ of hadron irradiation).

of atoms dislocation in materials which distinguishes hadron irradiation from γ-irradiation.
During the hadron irradiation various nuclear reactions like $^{12}C(\pi^-, \pi^- n)^{11}C$, $^{12}C(n, 2n)^{11}C$, $^{12}C(p, pn)^{11}C$ etc. go in the plastic samples mainly with the production of β^+-radioactive isotope ^{11}C with the half-life time $T_{1/2}$=20.4 min. Positrons from ^{11}C almost immediately annihilate with electrons of material producing of two 511 keV γ-quanta. Fig.18 shows the "decay curve" of the light output from PS based scintillation tiles samples caused by their induced radioactivity. One can see that PS based scintillators produce only short-lived radioactive isotopes with half-life time of 20.5 minutes which is very close to the tabulated value for ^{11}C isotope. The points corresponding to different accumulated doses nicely fit the same curve. It means that at our irradiation rate an "equilibrium" $creation \rightarrow decay$ is reached in less then 2 hours.
Data analysis demonstrate negligable effect of Atlas scintillation tile calorimeter degradation under irradiation at maximum luminosity.
The authors would like to thank A.Chumakov and V.Vasil'chenko for their help in samples irradiation and M.Tavlet (CERN) for the dose measurements with alanine dosimetry.

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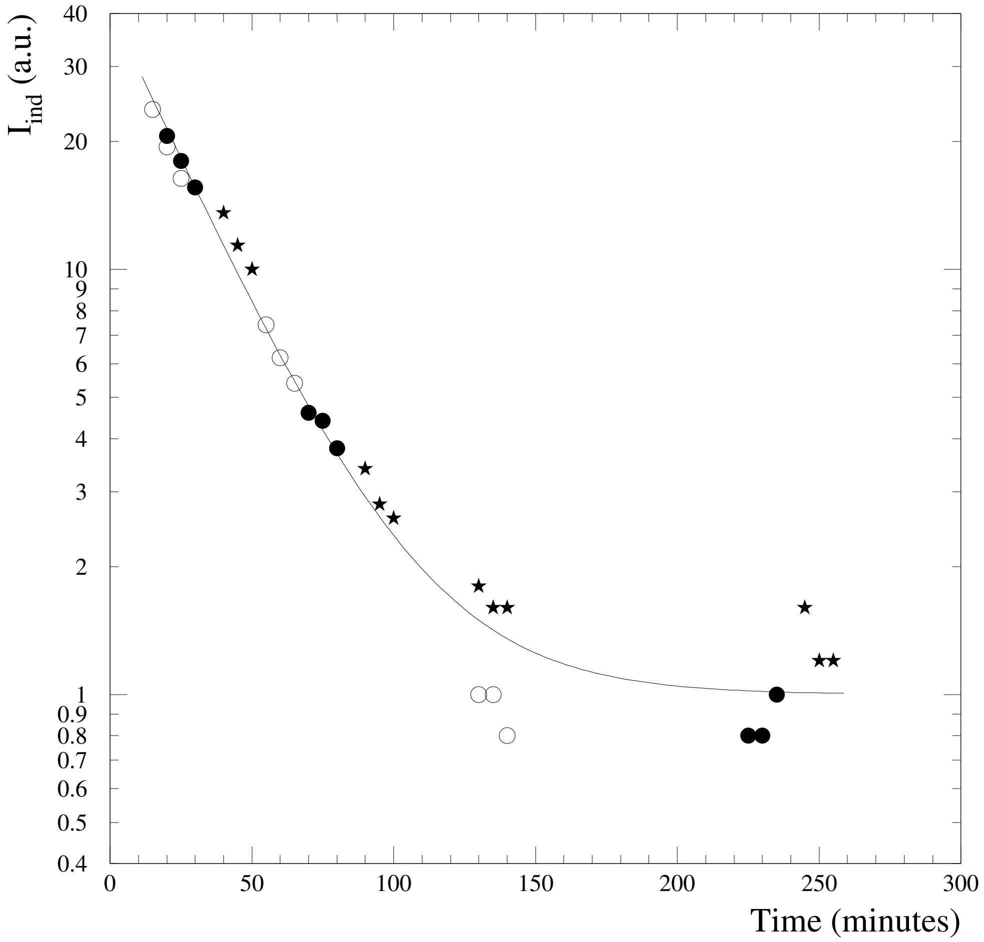

Figure 18: "Decay curve" of the light output from scintillation tiles caused by induced radiactivity after receiving different doses of hadron irradiation ($\bullet$–$0.24kGy$, $\circ$–$0.48kGy$ and $\star$–$1.68kGy$). $Fit\ =\ A * exp(-t/\tau) + B$.

Appendix

The list of dopants used in this study:

pTP – para-terphenyl, λ_{abs}=275 nm, λ_{em}=340 nm

PPO – 2,5-diphenyloxazole, λ_{abs}=310 nm, λ_{em}=365 nm

POPOP – 1,4-bis-[2-(5-phenyloxazolyl)]-benzene, λ_{abs}=365 nm, λ_{em}=420 nm

DMPOPOP – 1,4-di-2-(4-methyl-(5-phenyloxazolyl))-benzene, λ_{abs}=370 nm, λ_{em}=430 nm

BBOT – 2,5-di-(tret-butyl-2-benzoxazolyl)-thiophene, λ_{abs}=380 nm, λ_{em}=436 nm