Results of the gluing tests carried out in Rome from December 2000 and May 2001

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1. Curing time

Up to now three glues are resulted more promising for CMS: Histomount, NOA61 and DC3145. The curing time declared by the manufacturer is reported in Table 1, but it have to be verified in the operative conditions of CMS, that is by gluing PWO crystals with capsules. The curing time test is realised by gluing groups of 5 crystals at time (to have a minimal statistic), and testing the adhesion after the specified time. The adhesion test consists in hanging 80g on the capsule: if that does not cause ungluing along several weeks, the gluing at the considered curing-time is considered suitable for CMS purposes.

Table 1: declared and effective curing time for CMS

Glue	**Curing**	
	Declared	**Experimental**
Histomount	several hours	longer than 1 month
NOA61	3J/cm^2 @ 350-380 nm	6 min with 15mW/cm^2 @ 360± 25 nm
DC3145	72 hours	24 h

1.1 Histomount

Regarding Histomount, "several hours" of curing are absolutely not sufficient: although after some days the capsule seems well glued to the crystal, when the sample is submitted to the adhesion test, the complete separation of the capsule from the PWO occurs in one day about. The investigation of the curing time was extended till 1 month, never reaching positive results. On the other hand some samples glued on December 2000, and submitted on March to the adhesion test, resulted well glued. In any case several months of curing time is totally unacceptable for CMS.

As reported later, among the considered products, Histomount gives the highest light collection. In order to reduce the curing time, Histomount should be supported by another glue, just dedicated to the mechanical adhesion. Very interesting results have been obtained with a cyanoacrylate adhesive. Four capsules were prepared as follows: one drop of Histomount on the centre of each APD, and two thin strips of cyanoacrylate adhesive along the capsule frame. After 3 hours the samples were removed from the gluing bench and submitted to the adhesion test with positive result.

1.2 NOA61

NOA61 cures when exposed to UV light; the recommended energy for the full curing is 3J/cm^2 @ 350-380 nm. A collimated Cermax Xe lamp (175W) together with an UV filter centred at 360 ± 25 nm and a first face aluminium mirror are used. The UV filter avoids to damage PWO with UV out of the spectral range useful for the curing; the mirror makes easier the alignment of the UV light beam. The power flux measured with a calibrated photodiode is 15 mW/cm^2 at 40 cm far from the light source. The PWO//capsule interface (at the "rear face") is illuminated throughout the "front face", that is the UV light longitudinally crosses the crystal. We preferred this configuration respect to the lateral exposition because it allows to precisely determine the UV power flux reaching the glue and then the exposure-time needed for the curing. As a matter of fact, in order to by-pass the critical angle constrain, the UV should be injected through the de-polished lateral surface, but the evaluation of the UV energy reaching the glue layer is very difficult. Taking into account the typical longitudinal transmittance of full size PWO crystals, 6 minutes is the expected curing time with 15 mW/cm^2.

The UV exposure may damage crystals and detectors and this may be an argument against the use of NOA61. For several crystals we studied the induced damage by measuring the longitudinal transmittance before and after 6 minutes of UV (without gluing). Figure 1 shows the decreasing of the longitudinal transmittance, averaged on 400-700 nm range: soon after the UV exposure, it is about 0.5%; after 5 h the damage is completely recovered (the crystals were exposed to artificial light, the lamps of the laboratory). One crystal appears slightly better after the UV irradiation; probably at the beginning it had some defects, not recovered because the storage in the dark. We also studied the effect of the UV irradiation on the crystal light yield and the capsule sensitivity by submitting one sample, consisting in PWO and capsule not glued, to the x-ray test in different conditions: i) before any UV irradiation, ii) after the solely crystal irradiation, and iii) after the capsule irradiation. At least one day elapsed between each measure and the following one to ensure the crystal recovery; as a result none signal variation was observed.

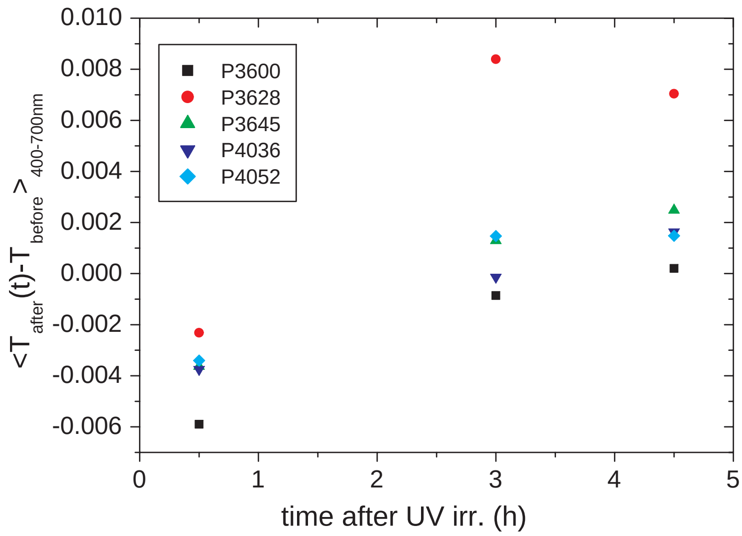

Figure 1: decreasing of the longitudinal transmittance, averaged on the range 400-700 nm, at different time after the UV irradiation of the crystal. The error is about 0.003.

Five samples were glued according with the above-specified conditions, and all passed the adhesion test. For two of them, after one week, a worsening of the optical contact between PWO and capsule frame was observed with the bubble-viewer, as if the glue was become again liquid. On the other hand, the adhesion between PWO and APD looked perfect. The capsule of one of these samples was separated from the crystal by mechanical torsion: the capsule frame and the corresponding part of the PWO surface were wet of liquid glue, but small pieces of PWO were on the APDs surface, witnessing the quite strong adhesion between APD and PWO. This partial failure cannot be attributed to cleaning, because the same procedure was applied to all the samples, or to under-valuation of the UV exposure, because the considered crystals have similar longitudinal transmittance; indeed it seems due to the low control of the glue-layer thickness ensured by the actual gluing procedures. As matter of fact, now, during the glue curing, capsule and PWO are compressed one to each other by means of the weight-force of the crystal, 1.1 Kg about. On the other hand, in order to ensure the best optical contact with the crystal, the APDs are elastically mounted in the capsule by a thrust bearing made of foam rubber, slightly jutting out the capsule frame (few tenths of millimetre). For several capsules we measured, without glue, the compression force at which the tops of the two APDs are at the same level of the frame; the value varies from capsule and capsule, ranging from 2 to 2.5 Kg. Therefore, probably the failure is occurred for those samples having the glue layer too thick to cure with the prescribed UV dose.

Anyway, the compression of capsule and PWO by the only weight force of the crystal, results not sufficient to completely flatten the capsule frame to the PWO surface; the thickness of the glue layer between capsule frame and PWO is not minimised and it has not the same value for all the samples. Basically there are two solutions for this drawback: to increase the compression force or soften the thrust bearing of foam rubber. The latter solution is more convenient, because reduces the risk of ungluing of the capsule frame from the crystal, and does not require any modifications to the gluing benches. Please note the thickness minimisation of the glue layer, including that between capsule frame and PWO, is desirable for each kind of glue, in order to improve the mechanical adhesion and reduce the effect of the aging.

1.3 DC3145

DC3145 cures by air moisture; the declared time for the full curing of a transversal section 3mm thick is 72h. Ten samples were glued and all passed the adhesion test just after 24 h of curing.

This glue is much more viscous than the other, therefore the imperfect minimisation of the glue layer thickness by the actual gluing procedure is more evident, but it has never caused the failure of the adhesion test.

2. Indirect contribution in recent APDs

The *bubble meter* is one of the tools proposed for the inspection of the gluing (Fig.2). It was designed and tested by means of early version of APDs, and full size PWO crystals with polished later surfaces. In the early versions, the envelope of Hamamatsu APDs was black, the active area circular, and the protection window made in silicon resin with concave surface. The LED case of the *bubble meter* was designed to make uniform the light intensity on the rear face of the crystal. In these conditions we experimentally observed a good correlation between the signal increment (before and after gluing) and the portion of the APD active area covered by air bubbles.

The gluing tests here reported were dealt with detectors and crystals of recent production: now the APD envelope is made of ceramic material, white and diffusive; the active area is squared; the protective window is convex and made of epoxy glue; the APDs are assembled two by two in capsule. Moreover, one of the four lateral surfaces of the crystal is grinded in order to make uniform the light yield along the longitudinal axis.

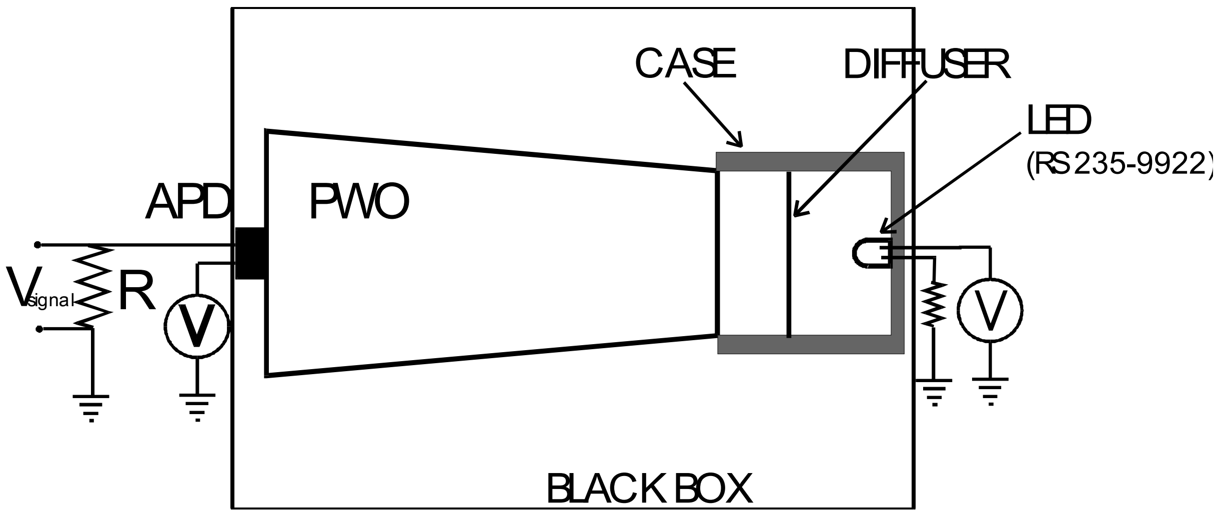

Figure 2: bubble meter

Two gluing benches used at the Rome Regional Centre are equipped with the *bubble meters* and they have been used for many of the samples here considered. The observed signal improvements are summarised in Table 2; they are not systematic and always less than the expected value, even if no sample was affected by air bubbles, thanks to the convexity of the APD window surface.

Table 2: predicted and measured signal improvement

glue	η_{theory}	$\eta_{experiment}$
Histomount	1.172	1.07 ÷ 1.15
NOA61	1.160	1.07 ÷ 1.09
DC3145	1.134	1.08 ÷ 1.11
grease	1.140	1.08 ÷ 1.11

We studied the problem with the earlier version of the *bubble meter* obtaining results very surprising. First, the intensity at the rear face of crystals with one lateral surface grinded is not very uniform, and then the readout depends on the position of the capsule. As a consequence, the right working of the *bubble meter* requires the precise repositioning of the capsule on the crystal surface, before and after the gluing.

Second, and more serious drawback: the signal induced by lightning new APDs (S) has a relevant indirect contribution ($S_{indirect}$) due to the photons impinging outside the active area, but detected after diffusion and reflection (see Fig. 3). The weight of the indirect contribution $S_{indirect}/S$ is reduced when the APD is facing the glue, and the amount of that decreasing varies from APD to APD, causing the bad working of the *bubble meter*. The indirect contribution $S_{indirect}$ was determined by comparing the signal measured without and with the masking of the APD entrance window around the active area; we found that $S_{indirect}/S$ is about 23% and 14% when the capsule is coupled to the PWO by air and optical grease, respectively. On the contrary in APDs with black envelope the indirect contribution is about 0.1%. We considered masks with different extension, and found that the most part of the indirect contribution is due to the photons crossing the protective window close to the boundary with the ceramic case.

It should be stressed that the indirect contribution improves the APD sensitivity, and than it is welcome, even if it makes the *bubble meter* useless. Perhaps Hamamatsu should be prompted to investigate the possibility to improve the direct contribution by modifying the internal walls of the ceramic envelope; we cannot study our self this argument because of the hardness of the ceramic material.

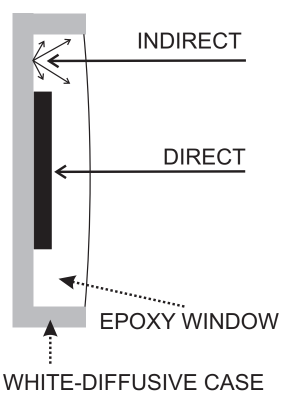

Figure 3: direct and indirect contributions in new APDs.

3. Glue optical effectiveness and optimisation of the PWO//capsule coupling.

The optical effectiveness of the glue is related to the ratio D/E, that is the ratio "number of photons detected by the capsule" to "emitted photons in PWO". The luminescence in PWO can be induced by ionising radiations, like x and γ rays; in ENEA-Casaccia both kinds of source are available: a. CHF 320G Gilardoni x-ray tube, at the National Institute of Metrology of Ionising Radiations, and a radioisotope ^{60}Co source (*Calliope* pool plant), respectively.

More precisely the optical effectiveness can be deduced by measuring the capsule current related to luminescence before (PWO//air//capsule) and after (PWO//glue//capsule) the gluing; the ratio of these currents is related to D/E

$$\eta = \frac{signal_{glue}}{signal_{air}} = \frac{(D/E)_{glue}}{(D/E)_{air}}$$

Accomplishing this measurement with different glues allows comparing their optical effectiveness:

$$\frac{\eta_{glue1}}{\eta_{glue2}} = \frac{(D/E)_{glue1}}{(D/E)_{glue2}}$$

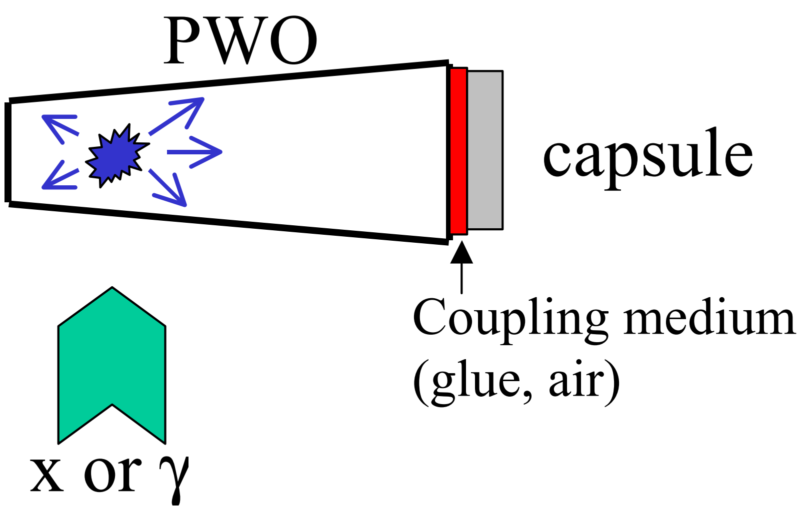

Figure 4: lateral irradiation of PWO crystals with ionising radiations.

Along 6 months, about 90 irradiations have been accomplished, initially with x and later with γ rays, according to the availability of the facilities. In both cases crystals were laterally irradiated (see Fig. 4) with a dose rate (in air) of about 10^{-3} Gy/s, giving a capsule current of few nA with 30 V supply (M~1, to avoid fluctuation of the APD gain). The current was measured with a pico-ammeter interfaced with a computer. Figure 5 shows the typical signal recorded during the irradiation; the recording includes some data before and after the irradiation to evaluate the dark current.

In the case of x rays, the ionising radiation is collimated in a beam having circular section (diameter 10 cm at 1 m far from the source) and intensity almost uniformly distributed. The energy spectrum of the x rays was pecked at 100 KeV for which the absorption length in PWO is 0.27 mm. The beam impinged laterally the crystal, centred at 15 cm from the rear face. The source is equipped with a mechanical shutter having driving time of few tenths of second; the samples were irradiated for 20s.

In the case of γ irradiation, the ionising radiations (1.17 + 1.33 MeV) is spherically distributed around the ^{60}Co source; the sample was placed 3.2 m far from the source, and shielded by a lead wall to reduce the dose rate at 10^{-3} Gy/s. At this energy the absorption length in PWO is 29.7 mm, therefore for lateral irradiation γ rays are not stopped in a superficial layer, and cross the crystal. Differently to the case of x rays, here the whole volume of the crystal is luminescent. The capsule with the terminal part of the crystal (about 2 cm) was further shielded to avoid the detector damaging. Crystals were wrapped with tyvek. The lifting and lowering of the ^{60}Co source in the pool takes about 1 minute and corresponds to the signal modulations bracketing the phase of steady irradiation, kept for 30s about (Fig. 5).

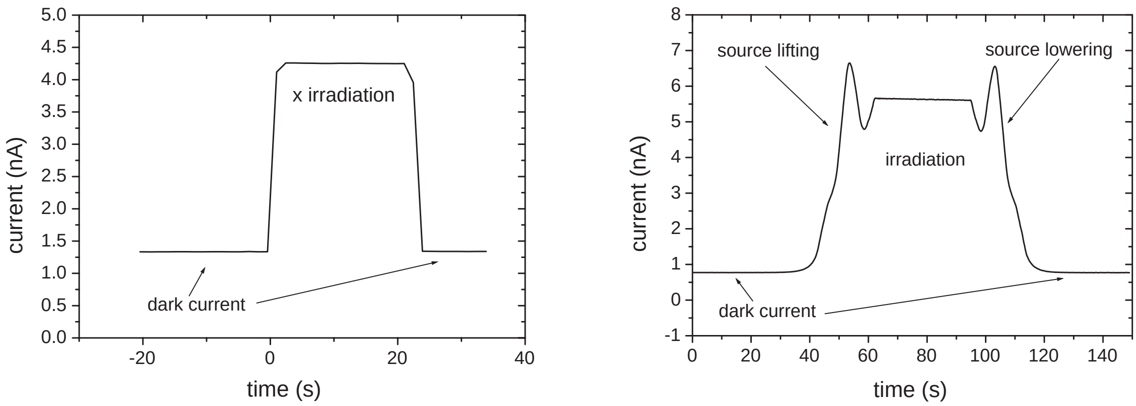

Figure 5: signal recorded during x and γ irradiations; the capsules were glued to PWO with DC3145.

For both x and γ irradiation, the signal versus time is slightly decreasing (few %) as effect of the crystal damage induced by the ionising radiations. To take into account the progressive damaging and the offset due to the dark current, the signal have to be processed as following: i) fit of the initial and terminal parts of the curve to evaluate the dark current present during the irradiation; ii) subtraction of the dark current; iii) fit of the part corresponding to the steady irradiation and evaluation of the current corresponding to the beginning of the steady irradiation; iv) correction of the obtained value for the temperature at which the irradiation took place, rescaling at 20 C. Analogously to the UV damage discussed in Section 1, the damage induced by both the irradiations is completely washed out after some hours, keeping the crystals exposed to artificial light (the lamps of the laboratory).

Each measurement session included a reference sample to check the reproducibility of the measurement; it is resulted better than 1%.

Table 3 summarises the results achieved in January and in March with x-ray irradiation, and compares the experimental results with the evaluation calculated on the basis of the optical constants of the involved materials; that calculus deals with an ideal interface PWO//glue//capsule (interface infinitely extended, PWO and Si semi-infinite). In the x-ray measurement, crystals were not wrapped to reduce the experimental uncertainty. Some others results are reported in Appendix.

Table 3: signal improvement by gluing and comparison of the optical effectiveness

Glue	η	η / η_{DC3145}	
		Experimental	Simulated
Histomount	2.450 ± 0.015	1.190 ± 0.008	1.192 ± 0.002
NOA61	2.19 ± 0.01	1.064 ± 0.007	1.159 ± 0.002
DC3145	2.058 ± 0.002	1	1

For Histomount the experimental value of η / η_{DC3145} is in perfect agreement with the simulated value.

NOA61 is superior (+ 6.4%) to DC3145, even if less than what expected (+15.9%). The reason of that disagreement is until now not clear: the optical characterisation of NOA61 was totally reconsidered but none error was found. In particular the refractive index was measured by the prism method; the transmittance was re-measured paying attention to exclude any effect for the possible glue luminescence.

In April and May further tests were accomplished by *Calliope* plant facility with the aim to widely study the argument. The achieved results are here summarised:

1) As already mentioned, the UV irradiation needed to cure NOA61 does not damage the capsule sensitivity.

2) The intensity distribution of the luminescence light outgoing from the rear face of the crystal is uniform, that is the light collection efficiency does not depend on the position of the APDs. This is not a trivial result, because the de-polished lateral surface of the crystal breaks the system symmetry.

3) The capsule frame, surrounding the APDs, back-diffuses the light escaping from the rear face; it improves the signal of 2% or 4% when unglued or glued (DC3145), respectively. Therefore the whole surface of the capsule frame should be glued to the crystal.

4) The indirect contribution to the signal obtained with new APDs (see section 2) glued to PWO with DC3145 is about 14%. The same value was observed with the *bubble meter* where light cross the PWO//APD interface at almost normal incidence; conversely luminescence light reaches the APD window also off normal incidence.

5) The evaluation of the optical effectiveness of NOA61 previously obtained with x-ray irradiation is confirmed with γ irradiation; it is less than the expected value: with respect to DC3145, about 1.06 instead of 1.159.

6) Grinding the PWO surface facing the APDs does not improve the signal.

7) The optical effectiveness of Histomount supported by a cyanoacrylate adhesive for the mechanical adhesion was evaluated on 4 samples. The results are quite spread, ranging from 0.97 to 1.162 (the expected value is 1.192), as a consequence of the high criticality of the glue dosage: Histomount should completely cover the APD window surface, avoiding the mixing with the cyanoacrylate adhesive that should be confined on the capsule frame. Therefore, to be adopted in CMS, this solution needs further study in order to optimise the dosage of the two glues.

4. Conclusions

The compression of capsule and PWO by the only weight force of the crystal (about 1.1 Kg), results not sufficient to completely flatten the capsule frame with the PWO surface; the thickness of the glue layer between capsule frame and PWO is not minimised and it has not the same value for all the samples. Basically there are two solutions for this drawback: to increase the compression force or soften the thrust bearing of foam rubber. The latter solution is more convenient, because reduces the risk of ungluing the capsule frame from the crystal, and does not require any modifications to the gluing benches. Please note, the thickness minimisation of the glue layer, also between capsule frame and PWO, is desirable for each kind of glue, in order to improve the mechanical adhesion and reduce the effect of the aging.

The *bubble meter* cannot be used to qualify the gluing, because the white envelope used in new APDs gives a relevant indirect contribution to the signal. This indirect contribution is due to photons impinging outside the active area, but detected after diffusion and reflection. The indirect contribution is reduced when the APD is facing the glue, and the amount of that decreasing varies from APD to APD, causing the bad working of the *bubble meter*.

Histomount is the glue giving the best optical effectiveness (+19% respect DC3145), but does not pass the adhesion test even after one month of curing; to be used in CMS it should be supported by another glue just dedicated to the mechanical adhesion. Very interesting results (until +16% with respect to DC3145) have been obtained with a cyanoacrylate adhesive, even if to be adopted in CMS, this solution needs further study in order to optimise the dosage of the two glues.

NOA61 cures in few minutes of UV irradiation; the required UV dose does not cause permanent damage to the clearance and the light yield of PWO, as well on the APD sensitivity. The minimisation of the thickness of the glue layer, also between PWO and capsule frame, is extremely important for the success of the gluing. Concerning the optical effectiveness, NOA61 is superior to DC3145 for +6%, but this value is less than the expected one; the reason is not clear.

DC3145 cures in 24 h, even when the glue layer is quite thick. Unsuccessful gluing was never observed. On the other hand, this product shows the lowest optical effectiveness among the considered products.

The capsule frame, surrounding the APDs, back-diffuses the light escaping from the rear face; it improves the signal of 2% or 4% when unglued or glued (DC3145), respectively. Therefore the whole surface of the capsule frame should be glued to the crystal.

APPENDIX: other results

A1. Signal improvement by wrapping

The wrapping of PWO crystals with diffusive or reflective foils improves the light collection; the x-ray facility allows to easily measure that improvement by measuring the signal without and with wrapping. Only tyvek wrapping was here considered, because metallic wrapping could affect the x-ray energy that is really absorbed by the crystal, causing an erratic evaluation. The table shows the considered optical system and the observed improvement factor.

Table 3: improvement factor by wrapping with tyvek

System	**Improvement factor**
PWO//air//capsule	2.66
PWO//air//PMT	2.53
PWO//grease//PMT	2.27

A2. PMT and capsule comparison

Concerning the optical configuration, the LY bench is quite different from the CMS operative conditions:

i) the PMT totally covers the rear-surface of the crystal and the remaining active area may be illuminated by the light emitted from the crystal lateral surfaces; conversely the active area of the capsule is just 0.5 cm^2;

ii) the material sequence is

PWO//grease//glass//metallic_layer and PWO//glue//epoxy_window//Si_3N_4_film//Si for LY and CMS, respectively. As a consequence both the critical angle and the detection versus the incidence angle are quite different.

Once again the x-ray facility allows to compare the signals obtained for these two optical configuration. To reduce the uncertain due to the PMT gain, we use a modified base at unitary gain. Analogously to the previous measure, the crystal was wrapped with tyvek. The experimental result is

$$\frac{Capule\_DC3145}{PMT\_grease} = 0.38$$

Gluing test collaborators:
Stefania Baccaro (irradiation expert), Giulio Capradossi (mechanics and gluing), Ioan Dafinei (crystal preparation and transmittance measurement), Giuseppe Ferrara (crystal preparation and curing time test), Sergio Guerra (electronics), Fabio Pellegrino (mechanics), Maria Pia Toni (X ray irradiation), Francesco Zarbo and Angelo Pasquali (γ irradiation).