DAPNIA/SED 95–01
CMS TN 95–032
6 February 1995

Study of some optical glues in a LHC-like environment

R. Chipaux, A. Soyer
CEA, DSM/DAPNIA/SED, CE-Saclay, France

Abstract

We report here the study of the transmittance properties of some optical glues and related compounds, of interest for the ETA and CMS scintillating crystal calorimeters, in the ultraviolet and after neutrons and photon irradiation.

Résumé

Nous rapportons ici les résultats d'une étude, dans l'ultraviolet et après irradiation neutronique et photonique, des propriétés de transmission de quelques colles optiques et produits assimilés, envisagés pour les calorimètres à cristaux scintillants d'ETA II et CMS.

1. Introduction

The interest to optimise the light collection in crystal calorimeters is obvious. The reduction of Fresnel reflections at the interface between the crystal and the photodetector needs to avoid any air gap. This could be obtain by the use of silicone gels or oils, or optical glues. This last possibility allows a mechanical binding as well. Many high density scintillating crystals have high refractive indices, (for example n = 1.95 for CsI, 2.15 for BGO, 2.2 for $PbWO_4$, at the wavelength (λ_M) of maximal emission), and it would be interesting, for a better optical adaptation, to use high index coupling materials.

The optical material used should of course transmit the scintillating light. This is not so easy when the light is in the ultraviolet range, like in CeF_3 or in pure CsI ($\lambda_M \cong 315$ nm), or even less in BaF_2 ($\lambda_M \cong 210$ nm). This material should also maintain its transmission properties during all the life of the detector, (specially for glues for which replacement is by definition impossible). That implies a low optical ageing, whether natural or under a high level of radiation. For example, at the shower maximum of the CMS electromagnetic calorimeter, the radiation level will be of the order of 10 kGy (1 Mrad) and 10^{13} fast neutrons/cm^2 [1].

The two aspects, ultraviolet transmission and radiation resistance, are not always mandatory in the same detector. $PbWO_4$, for example, which will be used in CMS, emits in the green range ($\lambda_M \cong 450$ nm). In the other way, pure CsI calorimeters, like proposed in the ETA II experiment at Saturne [2], would suffer a so important level of radiation, (CsI could not allow it, anyway), but a special care should be taken for the transmission of the ultraviolet light.

Previous published reports allow us to be rather confident to the possibility to find materials transmitting the ultraviolet (at least above 300 nm) and/or resistant up to more than 100 kGy, (or even 1 MGy in the visible range) [3]. Nevertheless these results have been obtained under gamma irradiation only, and other materials should be investigated, like the high index thermoplastic compounds from R.P. Cargille, Inc. We have undertaken, in the frame of the ETA and CMS projects, a study of the resistance of optical glues under “LHC-like”, i.e. hadrons + photons radiation environment.

We report here first tests performed with some optical materials commercially available : two epoxy resins, one acrylic resin, one silicone oil and one thermoplastic compounds.

2. Measurements

We followed a method similar to the one described in [3]. We used plates of fused silica, which dimensions (30 x 12 x 0.75 mm^3) were chosen to fit into the standard spectrophotometer sample holders. The plates were glued, two by two, following the process indicated by the manufacturers. The materials tested are listed in table 1.

Table 1 : Optical materials examined in this test.

Material	type	Manufacturer	Refractive index (at 589 nm)
E 501	epoxy resin	Epotecny	1.53
NOA 81	epoxy resin	Epotecny	1.56
Araldite 4001 uv	acrylic resin	CIBA	1.46
Meltmount 1.704	thermoplastic	R.P. Cargille lab.	1.70
Q2-3067	silicone oil	Dow Corning	1.46

All the samples and two unglued silica plates were irradiated in the channel HN1 of the Ulysse reactor. This nuclear reactor of the INSTN at Saclay provides radiation conditions close to the environment foreseen in the high energy detectors. We have already used it to study the radiation resistance of scintillating crystals and avalanche photodiodes, and its radiation spectra were characterised at this occasion [4]. Four irradiation steps were performed, whose characteristics are resumed in table 2. The cumulated fast neutron fluence is $1.0 \pm 0.2\ 10^{14}$ neutrons/cm^2 and the gamma dose (equivalent in air) 12.5 ± 4 kGy (1.25 ± 0.4 Mrad).

Table 2 : Characteristics of the irradiation steps.

date of irradiation	power (W)	duration (min)	fast neutrons fluence (n/cm^2)	cumulated	thermal neutrons fluence (n/cm^2)	cumulated	gamma dose (Gy)	cumulated
94/06/24	4.6	120	$8.0\ 10^{10}$	$8.0\ 10^{10}$	$2.3\ 10^{11}$	$2.3\ 10^{11}$	10	10
94/07/05	15	140	$3.0\ 10^{11}$	$3.8\ 10^{11}$	$8.8\ 10^{11}$	$1.1\ 10^{12}$	38	48
94/07/08	600	104	$9.0\ 10^{12}$	$9.4\ 10^{12}$	$2.6\ 10^{13}$	$2.7\ 10^{13}$	1100	1200
94/09/23	6000	104	$9.0\ 10^{13}$	$9.9\ 10^{13}$	$2.6\ 10^{14}$	$2.9\ 10^{14}$	11000	12500

The transmission spectra were recorded with a Perkin-Elmer Lambda 2 spectrometer, before irradiation and about two weeks after each step to allow the decrease of the induced radioactivity. The samples were not expose to strong UV light, including sunlight. Figures 1 to 6 show the transmission spectra as measured.

The two unglued plates were measured alone and coupled together with an optical liquid whose optical index dispersion matches quite well the dispersion of silica. Thus we accede to the equivalent loss of transmission due to the silica plates alone (i.e. Fresnel reflections at the air/silica interfaces and absorption in the silica). Then, neglecting the Fresnel reflections at the glue/silica interfaces and the absorption in the optical liquid, and assuming that all silica plates have the same transmission, we can estimate the intrinsic absorption of the glues. For a sample i, after a neutron fluence ϕ, if $Ts_i(\phi)$ and $Tp(\phi)$ are respectively the transmission of sample i and of the two joined plates, the intrinsic absorption $\rho_i(\phi)$ is given by : $\rho_i(\phi) = 1 - T(\phi)_i \cong 1 - Ts_i(\phi) / Tp(\phi)$. Figures 7 to 11 show the absorption of the different glues, converted for comparison to a thickness of 10 µm ($\rho_{i,10\mu m} = \rho_{i,x(\mu m)}^{(10/x)}$).

3. Results

For CsI, owing to their poor ultraviolet transmission, the thermoplastic compound and the silicone oil are rigorously not suitable, with an intrinsic transmission below 10 % at λ_M of CsI (see the figures and table 4). For the other compounds, the transmission at this wavelength is acceptable. However, we are just at the absorbency threshold, and the absorption would increase rapidly with the thickness of glue. The best material, E501, transmits for instance 99 % of the light through 10 µm, but only 76 % through 200 µm, at 315 nm. That would imply to limit the thickness of glue, and therefor to obtain the best possible crystal surfaces.

In general all tested glues except one are not affected by the neutron and gamma radiation, at least at the radiation level reached here and for the thickness of glue used. The exception is the two-compounds epoxy resin E 501, for which a degradation appears clearly above 10^{13} n/cm^2, (see figure 1). Nevertheless, taking into account the rather important thickness of this sample (~ 200 µm), the effect would remain weak for smaller thicknesses (see figure 7). An opposite behaviour is observed with the mono-compound epoxy resin NOA 81, which seems to improve under irradiation. However an ageing of at least 3 weeks is mentioned by the producer to allow the full reticulation of the resin, and the improvement observed could be partly due to that normal ageing. The small effects shown on figures 10 and 11 for the absorption of Meltmount 1.704 and Q2-3067 are not significant, because of the small thickness of these samples (10µm).

Table 3 : Summary of the results.

Material	Thickness	Wavelength for 50% and 90% transmission			
		before irradiation		after irradiation	
		50 %	90 % (90%/10 µm)	50 %	90 % (90%/10 µm)
E 501	~ 200 µm	301 nm	325 nm (297 nm)	316 nm	386 nm (297 nm)
NOA 81	~ 200 µm	380 nm	406 nm (357 nm)	367 nm	389 nm (313 nm)
Araldite 4001 uv	~ 200 µm	342 nm	376 nm (298 nm)	341 nm	376 nm (299 nm)
Meltmount 1.704	~ 10 µm	353 nm	377 nm	353 nm	377 nm
Q2-3067	~ 10 µm	347 nm	374 nm	349 nm	377 nm

Table 4 : Absorption through 10 μm of the studied materials at 315 and 450 nm (wavelengths of maximum emission for CsI and $PbWO_4$).

	Absorption at 315 nm		Absorption at 450 nm	
Material	before irradiation	after irradiation 4	before irradiation	after irradiation 4
E 501	1.0 % ± 0.2 %	3.7 % ± 0.9 %	≤ 0.02 %	0.25 % ± 0.07 %
NOA 81	14.7 % ± 3.4 %	9.6 % ± 2.3 %	≤ 0.02 %	0.06 % ± 0.02 %
Araldite 4001 uv	4.3 % ± 1.1 %	4.8 % ± 1.2 %	0.05 % ± 0.02 %	0.08 % ± 0.03 %
Meltmount 1.704	94.0 % ± 4.5 %	93.8 % ± 4.6 %	1.1 % ± 0.4 %	1.4 % ± 0.5 %
Q2-3067	89.4 % ± 6.7 %	90.3 % ± 6.3 %	0.7 % ± 0.4 %	2.5 % ± 0.8 %

4. Conclusion

This study is encouraging, in the sense that it is possible to find optical glues which transmit in the ultraviolet region, and are resistant to radiation levels up to at least 10^{14} fast neutrons/cm^2 and 10 kGy (1 Mrad). However attention should be paid to reduce the thickness of glue in pure CsI calorimeters. For lead tungstate crystals, the Cargille thermoplastic Meltmount 1.704 is a interesting compound, which allows reversible mounting, and has a high index of refraction and a good radiation resistance.

Acknowledgements

We are grateful to all the team operating the reactor Ulysse for their precious collaboration. We are indebted to J.-P. Peigneux, who gave us some of the samples, to J.-L. Faure, who initiated this study, and to P. Micolon for his constant support.

References

[1] G. Stevenson, in : *Proc. Large Hadron Collider Workshop, vol.3,* eds. G. Jarlskog and D. Rein, CERN/90-10, ECFA/90-133 (1990) 566-583.

CMS collaboration, *CMS Letter of Intent*, CERN-LHCC/92-3 (1992).

P.A. Aarnio and M. Huhtinen, Nucl. Inst. and Meth. A336 (1993) 98-105.

CMS collaboration, *The Compact Muon Solenoid, Technical Proposal*, CERN-LHCC/94-38, LHCC/P1 (1994).

[2] Saturne Proposition E284 ; see :
The ETA collaboration “Rare decays of eta meson”, in Nouvelles de Saturne, n°18, July 1994, p37.

[3] M. Kobayashi, S. Ishimoto, S. Sugimoto, S. Kobayashi, Nucl. Inst. and Meth. A305 (1991) 401-405.

[4] R. Chipaux, J.-L. Faure, P. Rebourgeard, G. Dauphin and J. Safieh, Nucl. Inst. and Meth. A345 (1994) 440-444 ;

R. Chipaux, J.-L. Faure, J.-P. Pansart, J. Poinsignon, P. Rebourgeard, J. Tartas, G. Dauphin, J. Safieh, “Study of neutron damage resistance of some scintillating crystals and associated photodetectors with the nuclear reactor ULYSSE”, in *Scintillator and Phosphor materials, M.R.S. symp. proc. v. 348,* (M.R.S., Pittsburg, 1994) p. 481-486, eds. M.J. Weber, P. Lecoq, R.C. Ruchti, C. Woody, W.M. Yen and R.-y. Zhu.

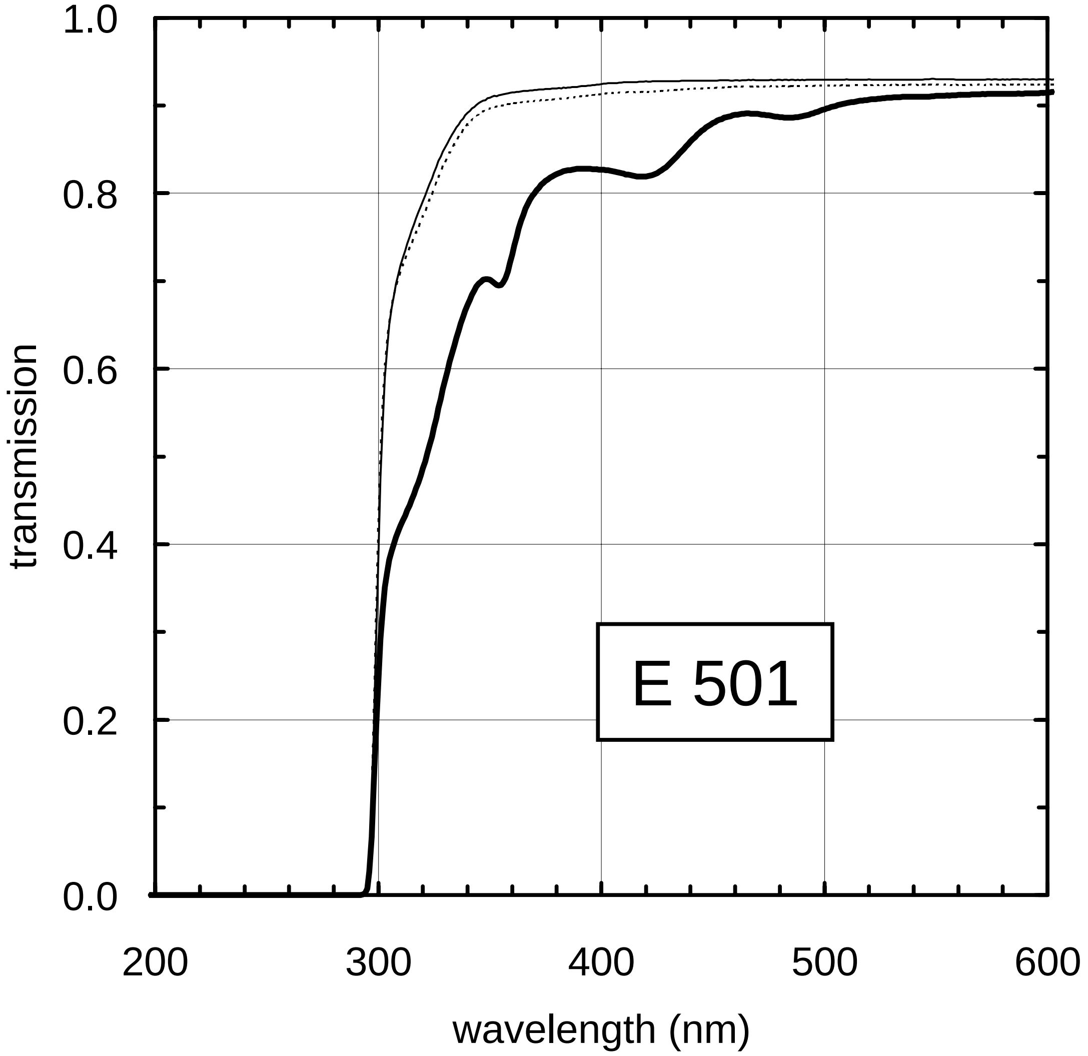

Figure 1. : Transmission spectra of E 501, thickness 200 µm, before irradiation (thin solid line), after irradiation 3 (~10^{13} n/cm^2, doted line), and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

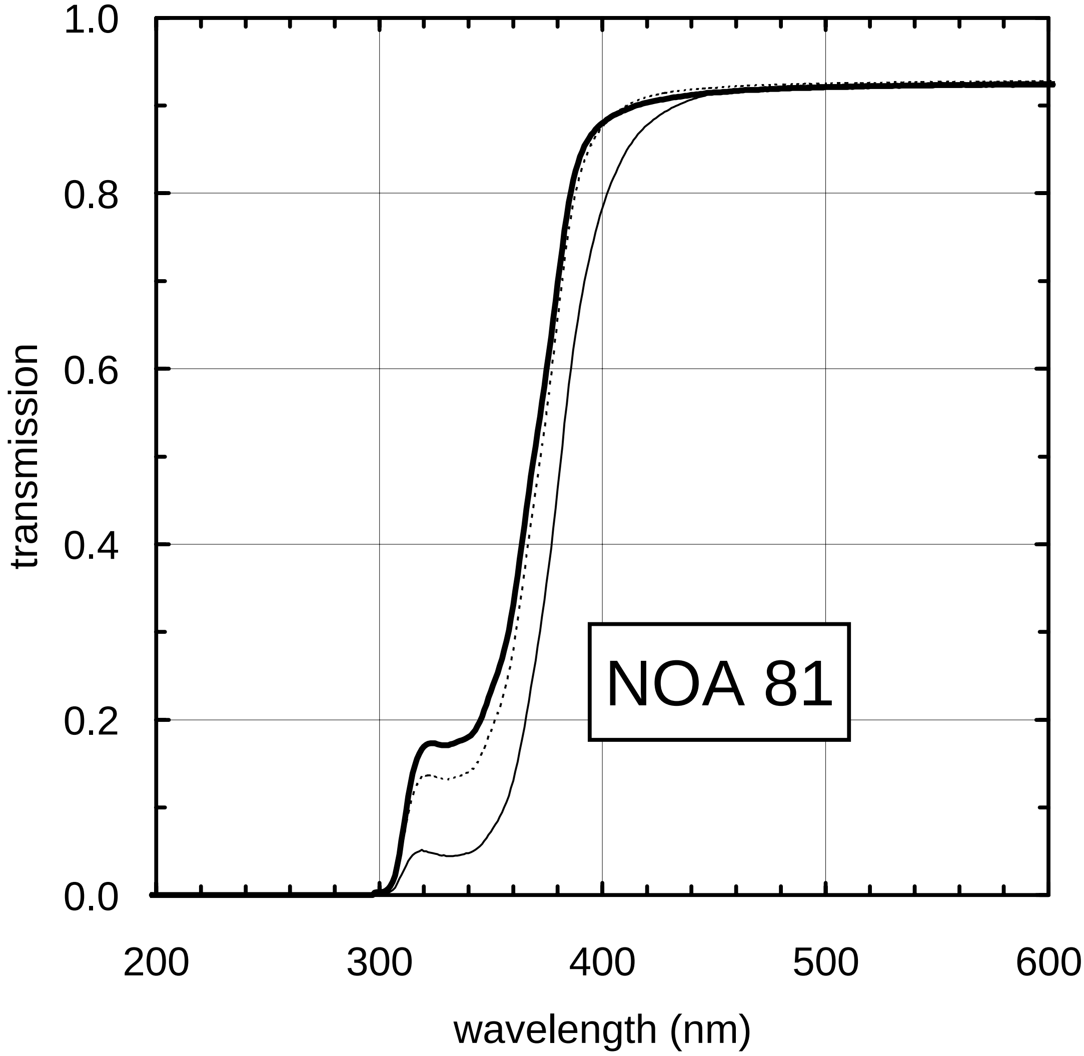

Figure 2. : Transmission spectra of NOA 81, thickness 200 µm, before irradiation (thin solid line), after irradiation 3 (~10^{13} n/cm^2, doted line), and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

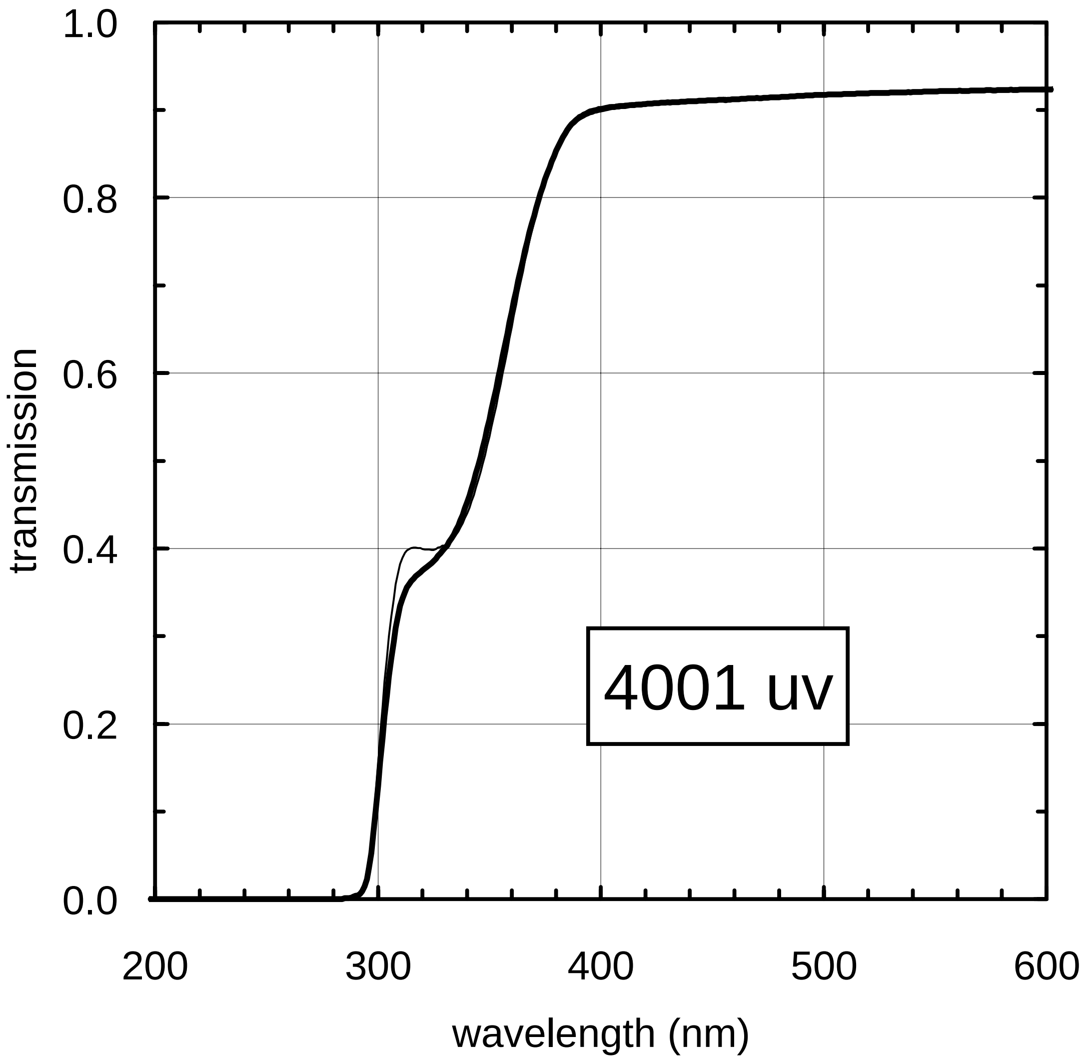

Figure 3. : Transmission spectra of Araldite 4001 uv, thickness 200 μm, before irradiation (thin solid line), and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

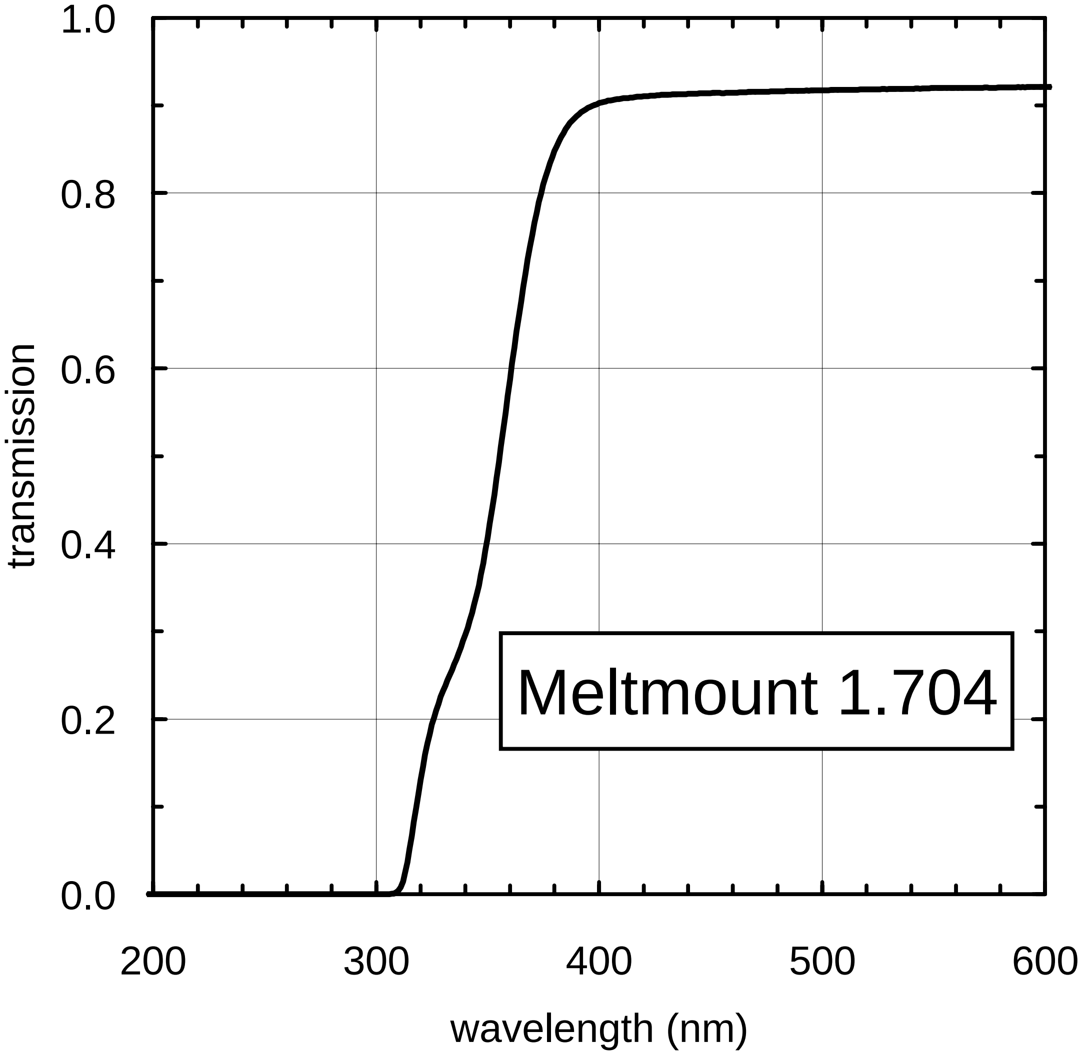

Figure 4. : Transmission spectra of Meltmount 1.704, thickness 10 µm, before irradiation, and after irradiation 4. The two curves are indistinguishable.

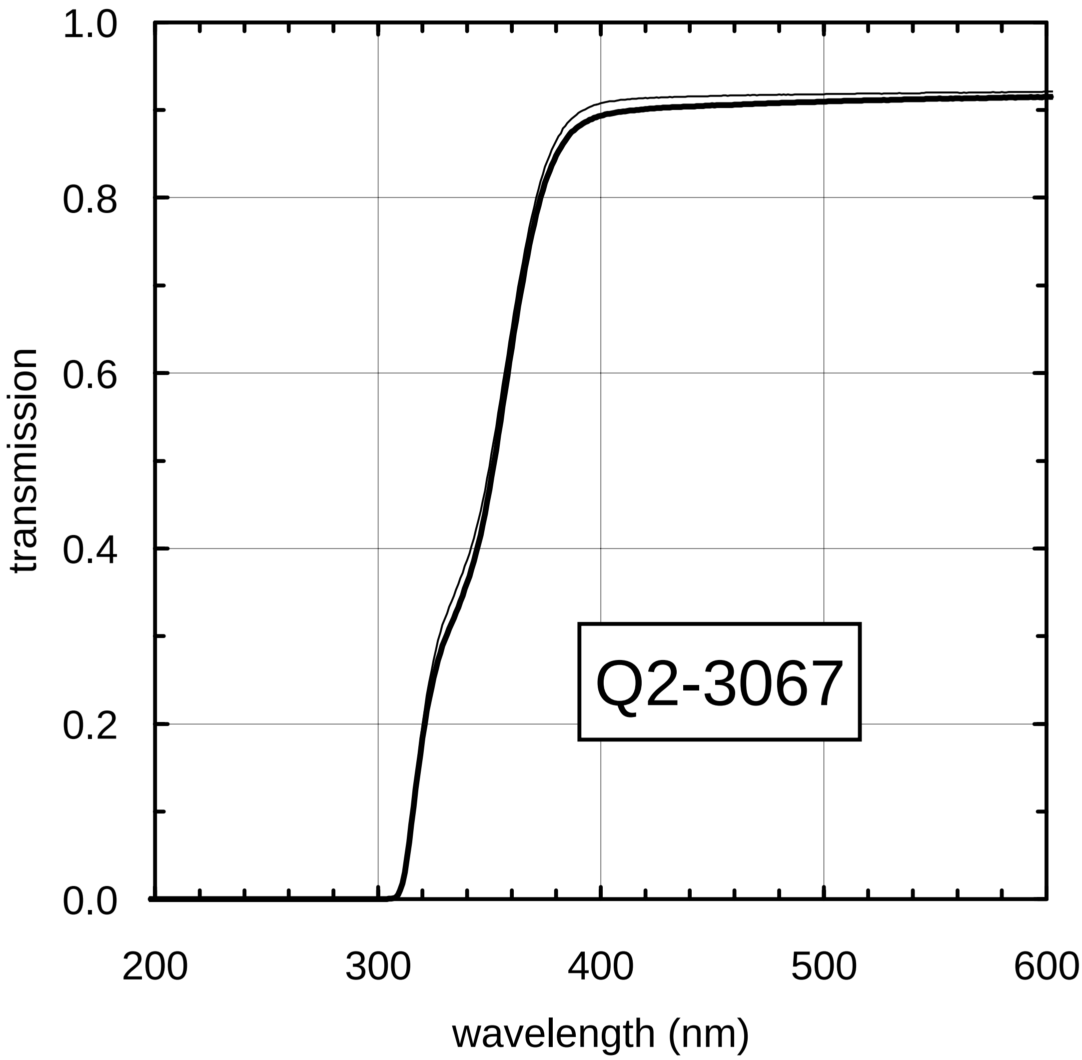

Figure 5. : Transmission spectra of Q2-3067, thickness 10 μm, before irradiation (thin solid line), and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

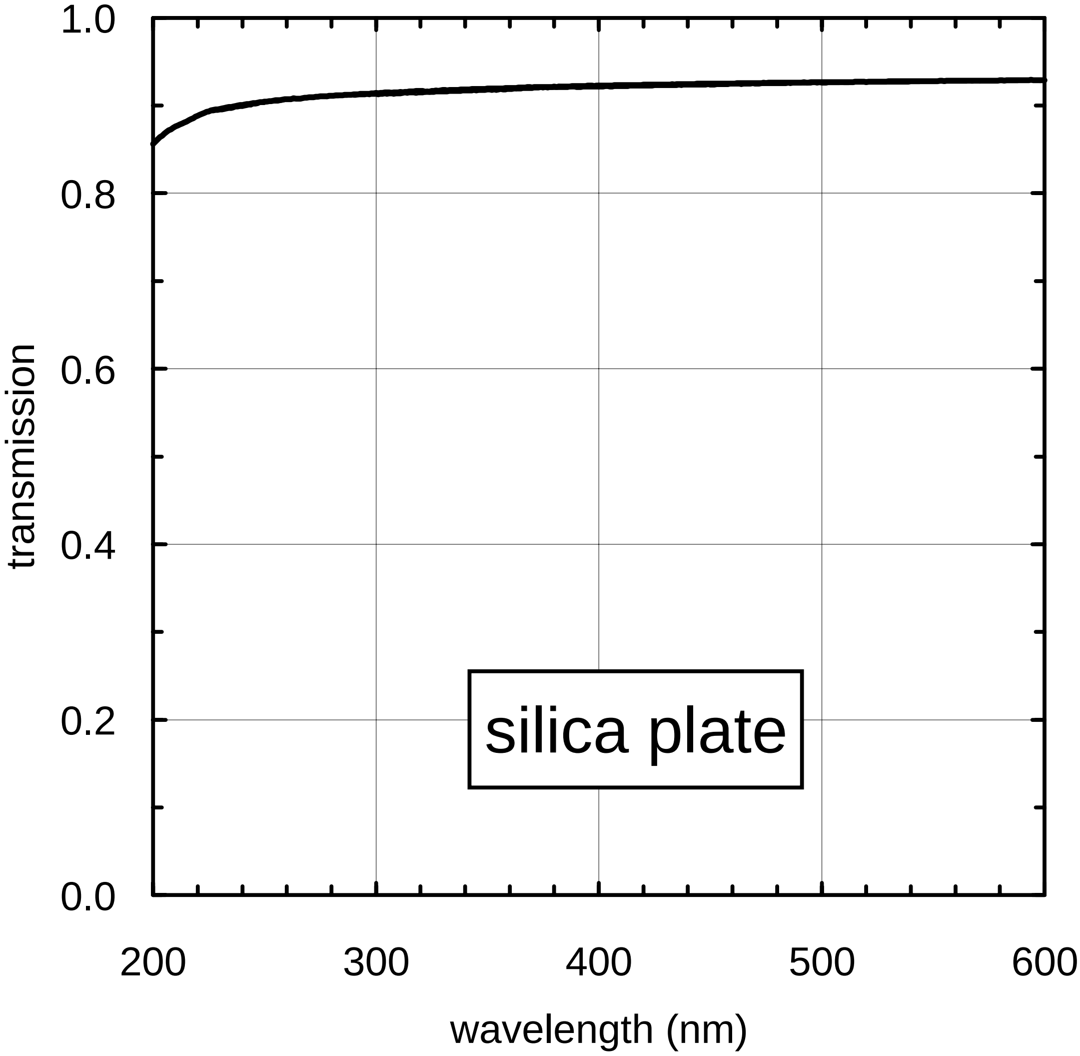

Figure 6. : Transmission spectra of one of the unglued silica plates, before irradiation and after irradiation 4 (~10^{14} n/cm^2). The two curves are indistinguishable.

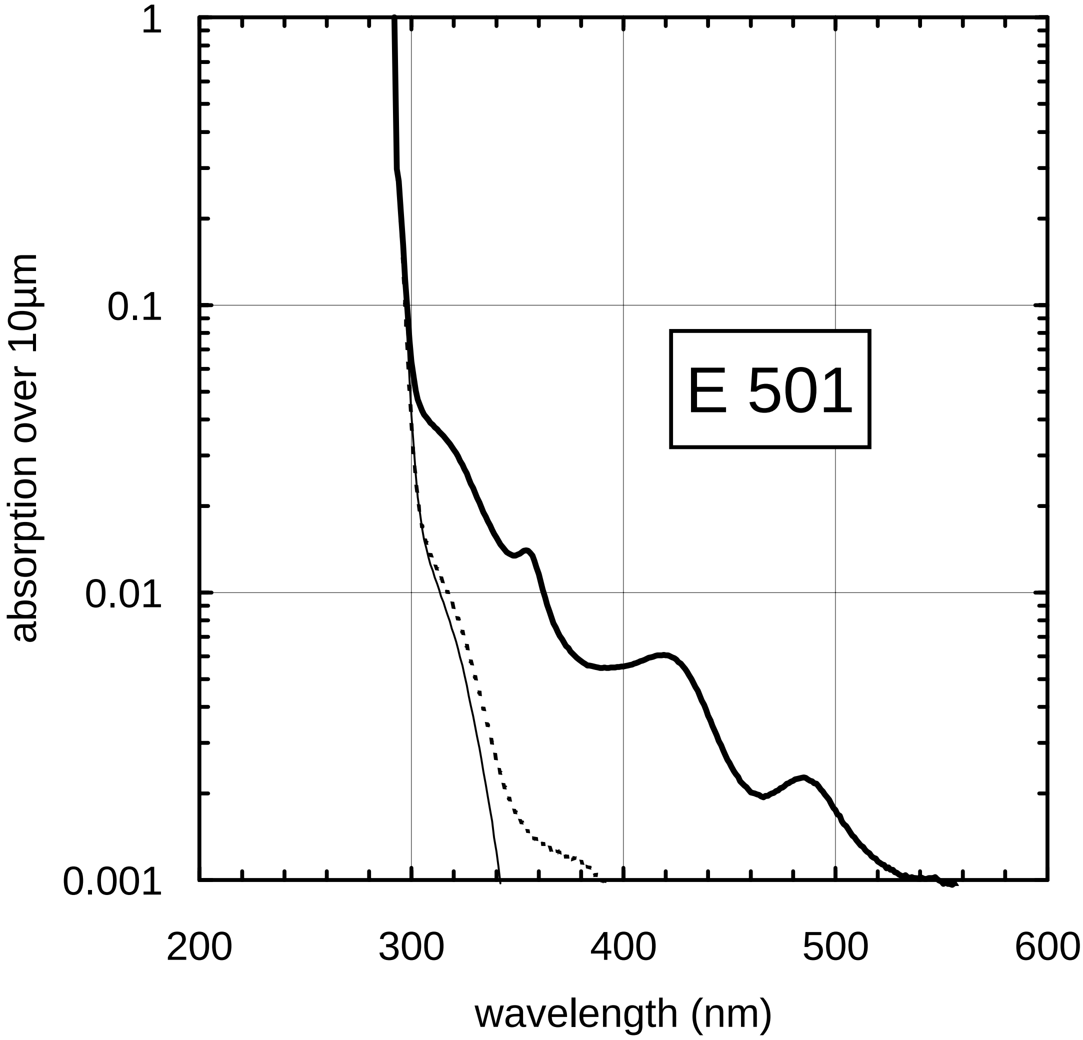

Figure 7. : Intrinsic absorption of E 501, calculated for a 10 µm thickness, before irradiation (thin solid line), after irradiation 3 (~10^{13} n/cm^2, doted line), and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

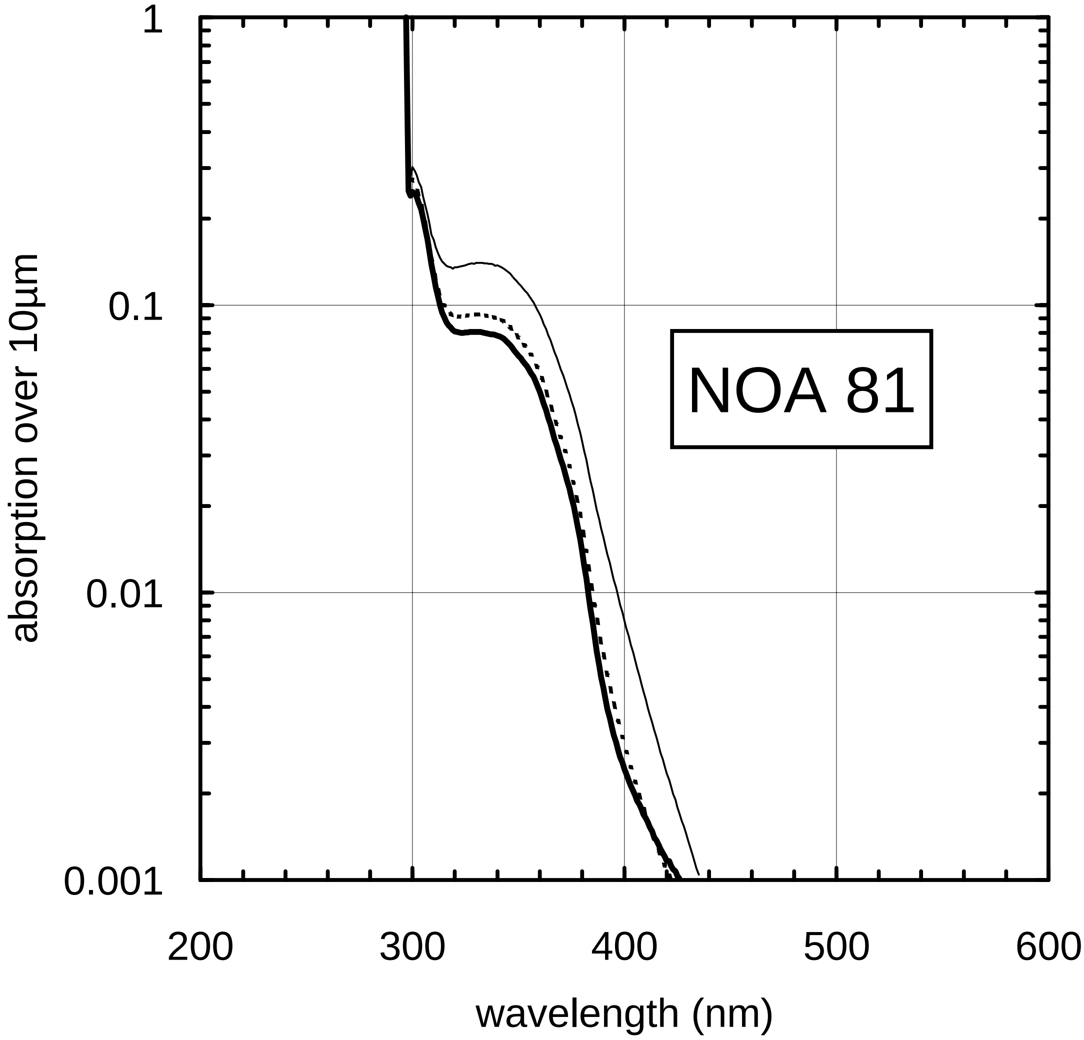

Figure 8. : Intrinsic absorption of NOA 81, calculated for a 10 µm thickness, before irradiation (thin solid line), after irradiation 3 (~10^{13} n/cm^2, doted line), and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

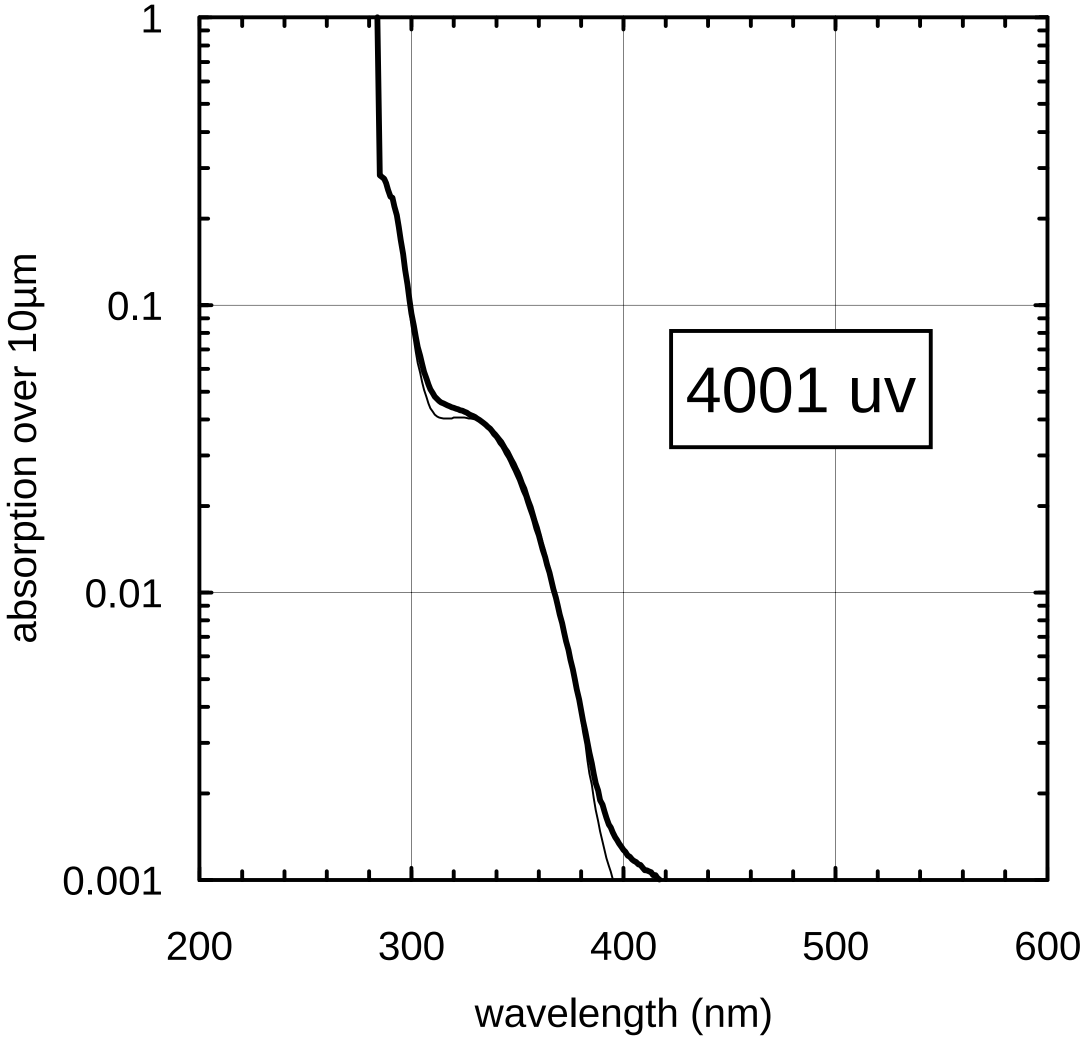

Figure 9. : Intrinsic absorption of Araldite 4001 uv, calculated for a 10 µm thickness, before irradiation (thin solid line) and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

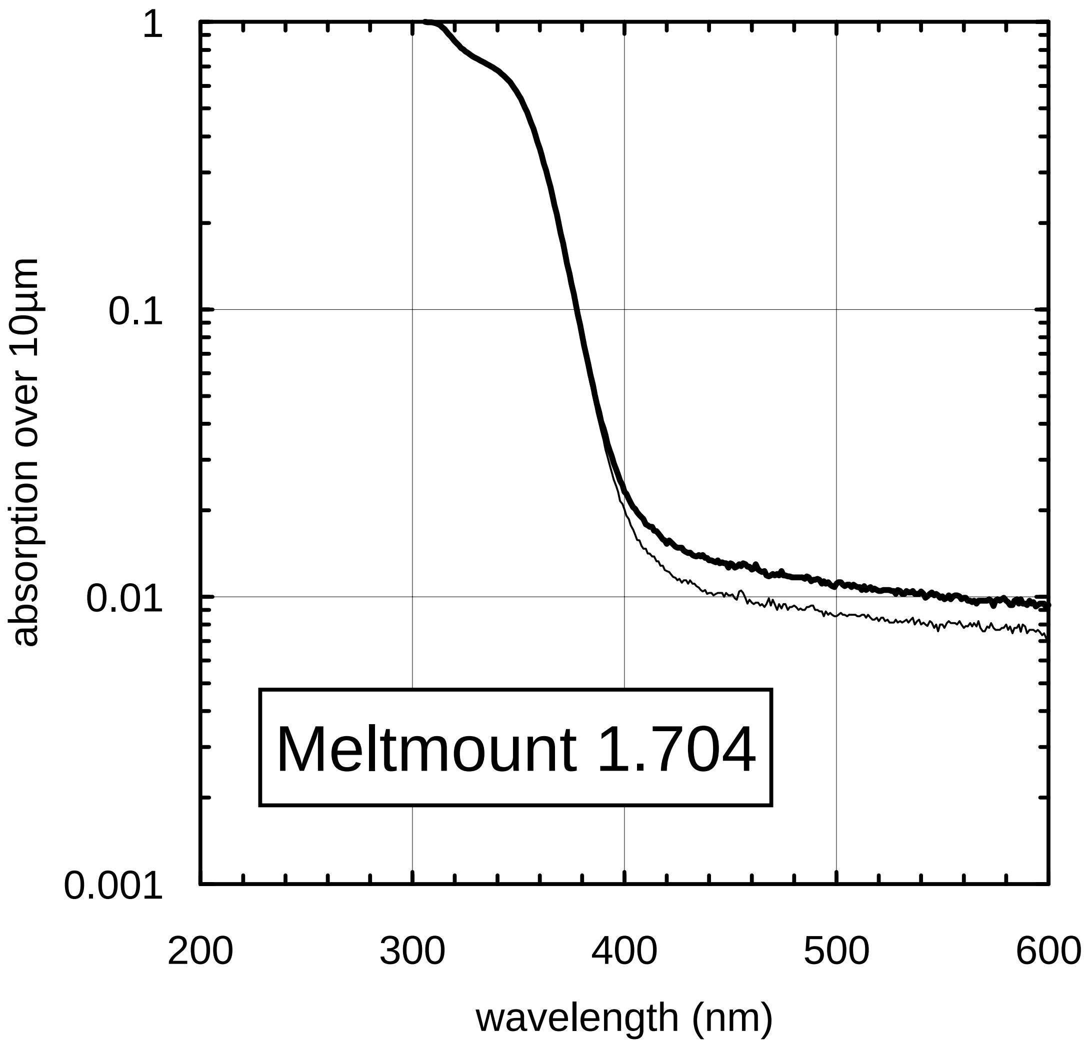

Figure 10. : Intrinsic absorption of Meltmount 1.704, before irradiation (thin solid line) and after irradiation 4 (~10^{14} n/cm^2, thick solid line).

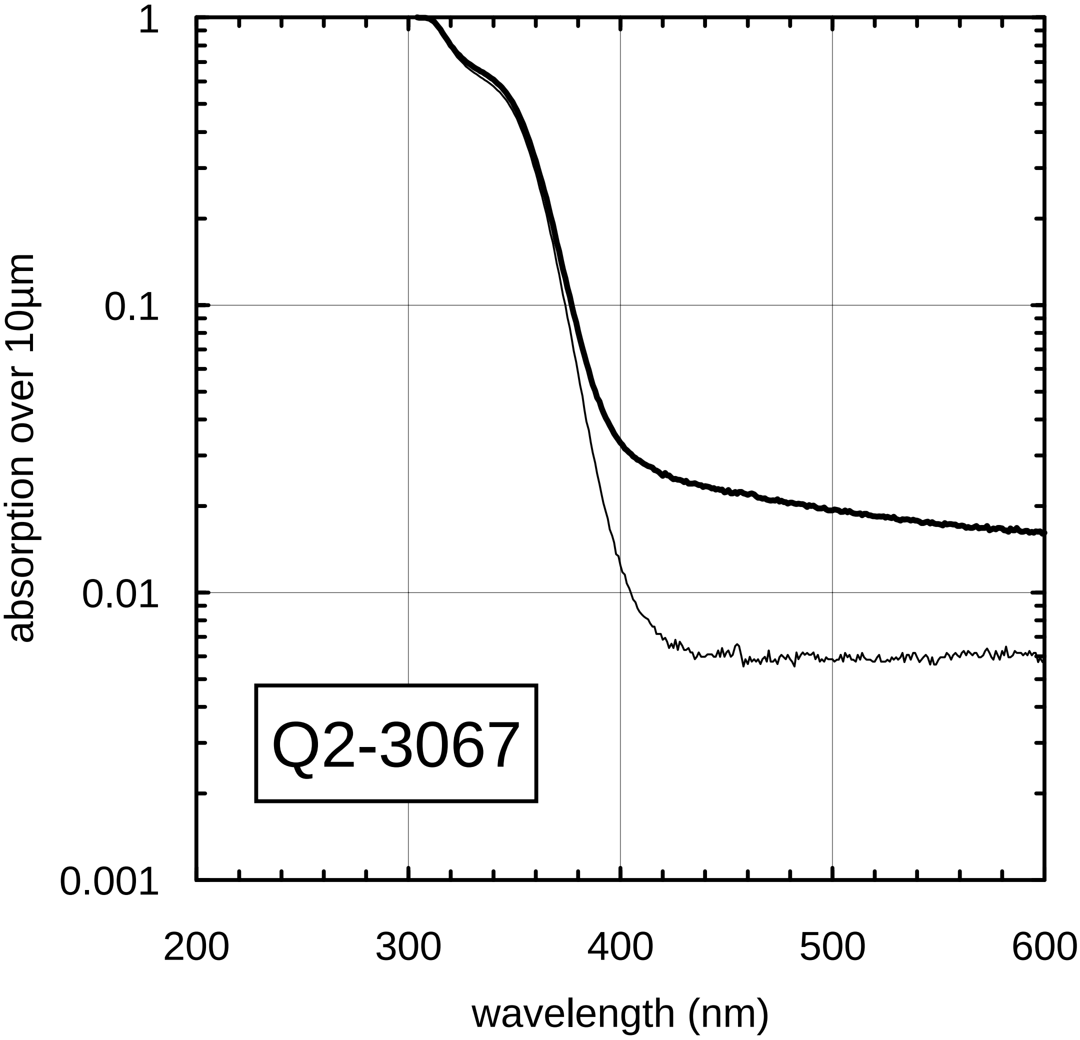

Figure 11. : Intrinsic absorption of Q2-3067, before irradiation (thin solid line) and after irradiation 4 (~10^{14} n/cm^2, thick solid line).