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**AGEING AT AMBIENT  
AND ELEVATED TEMPERATURES  
OF CARILON P1000 POLYMER**

by

**A. Lacroix**

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**AGEING AT AMBIENT AND ELEVATED TEMPERATURES  
OF CARILON P1000 POLYMER**

(July 1993 - September 1995)

by

**A. Lacroix**

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**SUMMARY**

Water absorption and specific mechanical properties of CARILON P1000 polymer viz tensile and Izod impact strength have been evaluated during one-year storage at ambient temperature.

CARILON polymer absorbs water at 23°C / 50% RH up to an equilibrium level of 0.45%. During storage, the tensile modulus increases, reaching a value of 1.8 GPa after one year. This effect appears to be completely reversible as annealing for 30 min at 60°C restores the initial value. This suggests physical ageing.

From the thermal ageing results the UL temperature index at 11,000 hours for the onset of embrittlement in tensile tests and notched Izod impact (half-strength value) have been determined. In both cases a temperature of 87°C was found.

November, 1995.

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**AGEING AT AMBIENT AND ELEVATED TEMPERATURES  
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## 1 INTRODUCTION

A major limitation on the life of polymeric components is the deterioration of their performance properties under service conditions.

For CARILON polymers it is known that both chemical (oxidation, cross-linking, chains scission) and physical ageing take place and its acceleration at elevated temperatures will lead to irreversible chemical changes affecting the morphological structure and hence the product properties. The distinction between chemical and physical ageing effects may therefore be difficult to make.

One method used in industry to quantify the effects of long-term heat exposure is the Temperature Index. It defines the (service) temperature at which a polymer loses 50% of a certain property after a given period of time. The property selected is considered the most critical for the application under consideration.

This programme is based on UL 746B standard (polymeric materials - long-term property evaluations) and will be carried out on the same polymer batch currently used in the IKV creep programme allowing a better analysis and interpretation of the results. Its aim is twofold: it establishes a room temperature storage base line for CARILON polymer P1000-1000 and it presents the influence of ageing at elevated temperatures on the properties of the polymer. Equally important are the properties measured by customers on the products received. They might use this for QC purposes and may also verify these findings at a later date. To cover an appreciable storage time properties have been monitored for more than one year, the results of which are discussed below.

## 2 THEORETICAL BACKGROUND

### 2.1 Physical Ageing

In semi-crystalline polymers, the origin of the physical ageing may be sought in the crystalline as well as in the amorphous phase. Consequently physical ageing of semi-crystalline materials in contrast with amorphous polymers occurs not only below but also above their  $T_g$ .

Below  $T_g$  the ageing is due to inherent instability of the amorphous glassy state. This process is also known as volume recovery, structural relaxation etc. Above  $T_g$  we can assume that crystallites disturb the amorphous phase and reduce segmental mobility. The consequence of this immobilization is that the glass transition is extended towards the high temperature side<sup>1</sup>. In contrast to chemical ageing, the physical ageing is a reversible process, i.e. by reheating the aged material at a temperature above the  $T_g$  the original state is recovered.

### 2.2 Arrhenius Equation

Thermodynamics determine whether a reaction is possible or not but the actual occurrence of a reaction depends on the kinetics. This in general is described by the Arrhenius equation<sup>2</sup>:

$$t = A \cdot e^{-\left(\frac{E_a}{RT}\right)}$$

with t: time (days)

A: constant

$E_a$ : Activation Energy (kJ/mole)

R: Gas constant (8.314 J/K)

T: Absolute temperature (K)

The time is determined as the time required to reach a specific end-point (for example 50% of an initial property) in a thermal endurance test. These tests are described according to different standards: UL 746B, IEC 216-1 or ISO 2578. In plotting the logarithm of the time as a function of the reciprocal temperature in Kelvin the activation energy can be derived from the slope. Subject to the life-expectancy the relevant temperature can be derived via graphical inter- or extrapolation. The times for the temperature index vary according to the method selected, viz. 11,000 hours for UL 746B and at 5,000 or 20,000 hours for IEC 216-1 (or ISO 2578) standards.

### 3 EXPERIMENTAL

CARILON polymer grade P1000-1000, batch 93/019 (LVN = 1.76 dl/g) was used for the evaluation. This batch was stabilised with additive Package 27 viz. 0.2% Calcium Hydroxy Apatite, 0.3% Nucrel 535 and 0.3% Irganox 1330.

Test specimens viz. tensile and Izod bars were injection moulded under standard conditions on a Demag 150-452 NC IIP equipped with the multi-cavity Campus mould without prior drying of CARILON polymer.

Details of injection moulding conditions can be found in Table 1.

Samples were evaluated on the basis of tensile strength and elongation (yield and break) following ISO 527/R at a rate of 50 mm/min. For the tensile modulus the chord modulus (0.2-0.3% strain) was chosen and determined at a rate of 1 mm/min. The notched Izod impact strength was determined according to ISO 180/1A. The number of test specimens was limited to 5 for tensile and 12 for notched Izod impact measurements.

After injection moulding, all specimens were conditioned in a vacuum oven in the dark at room temperature for one week; this treatment is defined as the DRY AS MOULDED (DAM) condition.

Following the above conditioning step room temperature storage was done in the dark in a black plastic bag at 23°C and 50% RH.

The water absorption to equilibrium was determined on the basis of the weight of 5 tensile and 5 Izod bars stored under the same conditions.

Up to reaching equilibrium of water uptake the selected properties were determined at shorter time intervals and after reaching equilibrium on a monthly basis.

Thermal ageing was carried out on the remaining samples in Precision Scientific ovens, model 625 set at 85, 100, 110 and 130°C. Verification of the temperature deviation exhibited a level of  $\pm 1^\circ\text{C}$ .

Table 2 provides an overview of the test regime, the number of specimens and the testing cycle.

## 4 RESULTS AND DISCUSSION

### 4.1 Ambient Temperature Storage

#### 4.1.1 Water absorption

Table 3 and Figure 1 show the average water content obtained by weighing 5 tensile and 5 Izod bars at 23°C and 50% RH in function of the storage time.

Storage time 0 h was defined as the time after completion of the conditioning in the vacuum oven (i.e. DRY AS MOULDED (DAM)) and it was assumed that the water content was close to 0%.

Water uptake to equilibrium was reached after a storage time of some 1000 hours (42 days). However, the equilibrium level was found to be lower for tensile (0.45%) than for Izod bars (0.50%). Assuming similar morphology for the tensile and the Izod bars water uptake at equilibrium should be more or less identical. The most likely explanation of this difference in water uptake can be found in the combined effect of the ratio surface area / volume of the specimen and the relative humidity which is accurate to  $50 \pm 5\%$ .

Because of the difference in dimension, the Izod bar has a larger surface area to volume ratio than the tensile bar, hence water uptake and loss is at a higher rate than that for the tensile bars. This can clearly be seen in Figure 1. However, when the samples are reaching equilibrium, the variation in absolute relative humidity starts playing an overriding effect. From the individual measurements, it was also noticeable that the tensile bars exhibited a much larger variability in incremental water uptake or loss. The most likely explanation for this phenomenon was that the specimen were not freely suspended but placed horizontally, hence not allowing free diffusion of water vapour into the non exposed area. In view of this observation, future measurements should be carried out with freely suspended specimen.

As can be seen in Figure 1, the water content decreased after around 120 days. This decrease by 0.1% corresponded to a drop in relative humidity level down to 40% RH caused by insufficient capacity of the air conditioning system during the very hot summer period. Consequently measures have been taken to correct this problem.

#### 4.1.2 Mechanical properties

Table 4 shows the effect of ambient temperature storage on the tensile and Izod properties of CARILON polymer.

The maximum tensile strength decreases from the DAM value of 60.7 MPa to an equilibrium value of around 59 MPa. The elongation at yield and at break both tend to decrease in function of the storage time: the former produces results of 22 to 19.5% while the latter only presents very scattered results with a tendency for an increase in standard deviation. The tensile modulus increases and level of 1.81 GPa was reached after one-year storage. A repeat measurement after some 2 years confirms this maximum level.

The notched Izod impact strength at 23°C increases from 16 KJ/m<sup>2</sup> to more than 19 KJ/m<sup>2</sup> in the first month before it reaches an equilibrium level of about 17 - 18 KJ/m<sup>2</sup> but at a larger standard deviation than initially found.

#### 4.1.3 Effect of water content on mechanical properties

It is well known from the literature<sup>3</sup> that water acts as a plasticiser, decreasing stiffness (modulus) in polymers and increasing their ductility. As it was shown in Figure 1, CARILON polymer absorbs water upon storage in humid air (23°C / 50% RH) which results in a weight increase of the specimen. However, as shown in Figure 2, this is accompanied by a slight plasticising effect up to the equilibrium in the water uptake. When the equilibrium is reached, the physical ageing, as demonstrated by the increase in modulus plays an overriding effect. These results are in line with the findings of KSLA work on flexural modulus<sup>4</sup>.

Nevertheless, as shown in Figure 3, the notched Izod impact strength increases during the first weeks of the experiment, suggesting plasticisation, but later on decreases accompanied with a larger scatter of results.

#### 4.1.4 Physical ageing

An increase in modulus upon storage at ambient temperature is also observed for other thermoplastics<sup>5,6</sup> and can be attributed to effects of a physical nature such as densification (free volume relaxation) and/or secondary crystallisation. In this case the increase in modulus is reversible upon a brief exposure to elevated temperature<sup>6</sup>.

This was verified on samples stored for 660 days in ambient temperature by annealing these specimen at a temperature of 60°C for 30 min in a vacuum oven. The tensile modulus dropped from a value of 1.81 GPa to a lower value of 1.34 GPa !, i.e. even lower than the originally measured level of 1.53 MPa. DSC measurements were performed before and after annealing but showed no effect on crystallinity as can be judged from the enthalpy value ( $\Delta H$ ) during the melting of the polymer.

The densification was not investigated.

The KSLA findings also showed that the increase of flexural modulus of CARILON polymer is reversible by annealing samples in the temperature range 40°C-140°C for 15 min in a vacuum oven purged with nitrogen<sup>4</sup>.

Judging these findings, it is considered most likely that physical ageing takes place and that a decrease in free volume in the crystalline-amorphous interface is likely to be the most important phenomenon involved<sup>7</sup>.

Above judgments are very relevant for elucidation to customers, measuring on the same product properties but at different time intervals, that physical ageing is the most likely explanation for differences observed. Also to keep in mind are differences in properties measured at other locations because polymers are tested after moulding (2 days minimum conditioning following ISO 291) and have not yet reached the water equilibrium level. The effect of water content on the mechanical properties can be important such as for Polyamides.

#### 4.2 Thermal Ageing

From the ambient temperature storage, it was concluded that a physical change occurs resulting in a significant increase in tensile modulus. However, it is known that at higher temperature physical ageing is overtaken by chemical ageing leading to irreversible chemical changes affecting product properties.

In a previous study<sup>8</sup>, thermal ageing up to 149 days was carried out on neat and coloured CARILON polymer.

In this program, extensive thermal ageing was carried out on CARILON batch 93/019 at four different temperatures, viz. 85, 100, 110 and 130°C according to the test regime shown in Table 2. The samples were evaluated on the basis of their tensile properties and notched Izod impact strength at 23°C after cooling down of the samples (around 1 hour after removing them from the oven).

##### 4.2.1 Results

The progression of the maximum tensile strength, elongation at yield, elongation at break, tensile modulus and notched izod impact strength for CARILON polymer batch 93/019 are presented in Tables 5 and 6 and in Figures 4 to 8.

Compared to the unaged sample, the tensile strength at yield increases by 10 MPa within 3 to 7 days ageing in function of the temperature. Upon prolonged ageing a continuing increase in yield strength is observed until the onset of embrittlement, corresponding to the loss of capability to yield. After the embrittlement, the tensile strength decreases rapidly. The elongation at yield decreases with time. The higher the ageing temperature, the more it decreases. No good explanation can be found for the increase to 24% at the highest temperature of 130°C.

The tensile modulus for all temperatures increases with time. Considering the Izod impact it can be seen that with time a reduction is apparent and taking place at higher rate at higher temperatures.

#### 4.2.2 Determination of temperature indices

In principle three criteria can be used to determine the temperature indices. The onset of embrittlement in tensile testing, 50% retention in tensile strength and half-value of izod impact strength can be considered.

The relation between ductility (samples yielding) and ageing time at different temperatures is shown in Figure 9. As criterion for loss of ductility the time for 50% of the samples to lose their ductility was calculated by regression analyses and resulted in 15 days, 63 days and 153 days for respectively at 130°C, 110°C and 100°C. No embrittlement was observed at 85°C within the current time frame.

A linear regression of these experimental data resulted in the following equation:  
 $\ln t \text{ (days)} = -26.02 + 11577/T \text{ (K)}$  with a coefficient of regression  $R^2$  of 0.9988. The activation energy calculated from the slope of this equation is 96 KJ/mole. Extrapolation to embrittlement at 85°C resulted in a period of 545 days.

Assessing on the basis of 50% retention in tensile strength was also performed. The initial tensile strength, necessary for determining the 50% retention value, was defined as the strength measured after 14 days ageing at the lowest ageing temperature, i.e. 70 MPa. The tensile strength of 35 MPa was achieved after 20 days at 130°C and at around 100 days (extrapolation) at 110°C. At lower temperatures, no further data were available to complete this analysis.

The same analysis was also made on the basis of the reduction of the Izod impact strength value. The initial value was set at 16 KJ/m<sup>2</sup> and 50% reduction of this value at each temperature results in a time of 17 days at 130°C, 59 days at 110°C and 176 days at 100°C. The 50% reduction of this property was not achieved at 85°C within the current time frame. These results are very similar to these based on the onset of embrittlement.

A linear regression in these experimental data resulted in the following linear equation:  
 $\ln t \text{ (days)} = -25.65 + 11458/T \text{ (K)}$  with a coefficient of regression  $R^2$  of 0.9825. The activation energy calculated from the slope of this equation is 95 KJ/mole. Extrapolation of the onset of embrittlement at 85°C results in a period of 568 days. Hence a time not differing greatly from that calculated for loss of ductility.

#### 4.2.3 Thermal endurance

A thermal endurance graph called Arrhenius graph is obtained by plotting the logarithm of time to reach a specified end-point in an ageing test as a function of the reciprocal absolute temperature. A thermal endurance graph based on the onset of embrittlement in tensile test, tensile strength and impact half-values is drawn in Figure 10.

The temperature index (T.I.) is the figure corresponding to the temperature derived from the thermal endurance graph at a period of time i.e. 11,000 hours for UL 746 B and 5,000 or 20,000 hours for IEC 216-1 or ISO 2578. After 5,000, 11,000 and 20,000 hours the T.I. determined on the basis of both the onset of embrittlement in tensile test or the impact half-value is respectively 96, 87 and 80°C. These temperature indices are in the same range as those indicated in the previous study<sup>8</sup>.

**5 CONCLUSIONS**

- . Water content at equilibrium of CARILON P1000 polymer determined at 23°C / 50% RH by weight measurement is about 0.45%.
- . Ambient temperature storage performed up to one year shows a significant increase in the tensile modulus which could be explained by physical ageing. A reversible effect was obtained by annealing the material 30 min at 60°C.
- . Temperature indices were determined from the thermal ageing data. UL temperature indices at 11,000 hours based either on the onset of embrittlement in tensile test or on the notched Izod impact strength half-value are 87°C.
- . IEC indices at 5,000 and 20,000 hours equal 96 and 80°C.

**6 RECOMMENDATIONS FOR FURTHER WORK**

- . Room temperature storage on specimens placed on a desiccator to better distinguish the effect of water uptake and physical ageing.
- . Investigation of thermal ageing under anaerobic conditions in order to better understand the different processes involved in the matter of chemical ageing.

Louvain-la-Neuve, November 1995.

## References

1. Encyclopedia of Polymer Science and Engineering, Ageing physical, vol. 1, p 595-610
2. D. W. Van Krevelen, "Properties of Polymers: correlations with chemical structure", Elsevier, 1972
3. BASF A.G., "Ultramid", technical brochure B568f, ludwigshaven, 1993
4. R. Hoff and F.J.M. Schnitzeler, "Influence of room temperature ageing on the flexural modulus of CARILON Polymer", AMRS 94.04
5. L.C.E. Struik, "The long-term physical ageing of polypropylene at room temperature", *Plastics and Rubber Processing and applications* 2 (1982) 41-50
6. L.C.E. Struik, "Physical ageing in amorphous polymers and other materials", delft, 1977
7. A.R. Postema and A.R.L. Leroy, "The long-term physical ageing of CARILON -EP at room temperature", RNO PT 018395
8. H.G. Kormelink, "Evaluation of colour masterbatches for CARILON polymer, including thermal ageing", LVGR.95.032

	Tensile	Izod
<b>Temperatures (°C)</b>		
Mould	60	60
Melt	234	234
Nozzle	240	240
Zone 1	240	240
Zone 2	240	240
Zone 3	230	230
<b>Times (s)</b>		
Injection	1	1
Hold	10	10
Cool	10	10
Cycle	27	26
<b>Pressures (bar)</b>		
Injection	80	80
Hold	70	65
Back	5	5
Cavity	335	335
Screw speed (rpm)	40	40

**Table 1:** Injection moulding conditions of CARILON polymer on the Demag D150-452 equipped with a multi-cavity Campus mould

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TEST	SPECIMEN		CYCLE
	Tensile bars	Izod bars	
Water absorption	5	5	
Dry as Moulded	5	12	
Ageing at 23°C, 50% RH for 1 year	85	204	monthly
Ageing at 85°C for 1 year	60	144	28 days
Ageing at 100°C for 6 months	65	156	14 days
Ageing at 110°C for 3 months	65	156	7 days
Ageing at 130°C for 1-2 months	55	132	3 days

**Table 2:** Overview of tests / specimen and cycle

		Water content, % weight	
		Tensile bar	Izod bar
Ageing time, h (days)	0 (0)	0	0
	24 (1)	0.091	0.116
	48 (2)	0.134	0.167
	72 (3)	0.157	0.194
	96 (4)	0.172	0.222
	168 (7)	0.219	0.275
	192 (8)	0.230	0.287
	216 (9)	0.250	0.317
	240 (10)	0.276	0.339
	264 (11)	0.293	0.360
	336 (14)	0.302	0.367
	408 (17)	0.322	0.386
	432 (18)	0.317	0.380
	504 (21)	0.335	0.400
	528 (22)	0.343	0.405
	552 (23)	0.368	0.434
	576 (24)	0.381	0.449
	600 (25)	0.388	0.455
	672 (28)	0.377	0.439
	744 (31)	0.408	0.470
	842 (35)	0.410	0.470
	1008 (42)	0.442	0.502
	1176 (49)	0.441	0.499
	1344 (56)	0.425	0.478
	1848 (77)	0.441	0.497
	2016 (84)	0.440	0.489
	2184 (91)	0.439	0.493
	2688 (112)	0.415	0.467
	3024 (126)	0.329	0.372
	3360 (140)	0.377	0.429
	3696 (154)	0.405	0.457
	3864 (161)	0.421	0.472
	4032 (168)	0.424	0.477
	4440 (185)	0.437	0.491
	4728 (197)	0.424	0.476
	5376 (224)	0.415	0.464
	6096 (254)	0.426	0.474
	6576 (274)	0.425	0.477
	8424 (351)	0.421	0.472

**Table 3:** Water content (% weight) at 23°C / 50% RH of CARILON 93/019 polymer

Temperature °C	Time days	Tensile strength at yield MPa	Elongation at yield %	Tensile strength at break MPa	Elongation at break %	Tensile modulus <sup>a</sup> GPa	Notched Izod impact strength KJ/m <sup>2</sup>
23	0	60.7 (0.3)	21.9 (0.1)	58.8 (21)	325 (42)	1.53 (0.03)	16.1 (0.3)
	3	59.3 (0.3)	22.1 (0.4)	35.5 (21)	221 (91)	1.50 (0.02)	18.3 (0.6)
	7	59.0 (0.4)	21.6 (0.4)	57 (15)	310 (41)	1.48 (0.02)	18.8 (0.6)
	14	59.0 (0.3)	21.6 (0.3)	57.9 (19)	305 (65)	1.47 (0.03)	20.4 (0.8)
	21	58.5 (0.1)	21.8 (0.7)	24.5 (22)	150 (95)	1.51 (0.06)	19.6 (0.8)
	28	58.1 (0.2)	21.9 (0.4)	48.7 (25)	283 (94)	1.44 (0.07)	18.8 (0.7)
	56	58.5 (0.3)					19.2 (0.8)
	67	58.4 (0.2)	20.8 (1.1)	55.3 (21)	270 (104)	1.51 (0.03)	17.5 (0.9)
	84	58.4 (0.3)	21.7 (0.5)	40 (27)	205 (131)	1.56 (0.04)	18.0 (1.1)
	112	58.9 (0.1)	22.6 (0.5)	25.7 (13)	127 (53)	1.49 (0.09)	17.9 (1.6)
	140	59.0 (0.2)	21.5 (0.4)	36.5 (20)	241 (76)	1.52 (0.07)	16.9 (1.3)
	164	59.0 (0.2)	20.8 (0.2)	43.1 (27)	236 (109)	1.67 (0.03)	15.4 (0.9)
	197	58.8 (0.3)	21.4 (0.4)	39.3 (30)	206 (131)	1.65 (0.02)	16.3 (1.1)
	224	58.7 (0.1)	21.0 (0.3)	25.5 (19)	145 (57)	1.63 (0.06)	15.7 (1.6)
	254	58.9 (0.2)	20.9 (0.3)	22.2 (10)	171 (55)	1.56 (0.03)	
	288	58.9 (0.2)	20.7 (0.2)	37.7 (25)	217 (124)	1.63 (0.06)	17.2 (0.5)
	319	59.3 (0.1)	20.3 (0.4)	10.6 (3)	98 (32)	1.72 (0.02)	15.8 (1.4)
	351	59.3 (0.1)	19.8 (0.1)	12.1 (10)	166 (85)	1.81 (0.08)	17.1 (1.3)
	±660	59.2 (0.3)	19.6 (0.08)		268 (83)	1.84 (0.02)	18.4 (1.2)

<sup>a</sup> = Chord modulus determined between the limits of 0.2 and 0.3% strain

( ) = standard deviation

' = Dry as moulded

**Table 4: Effect of ambient temperature ageing on tensile properties and notched Izod impact strength for CARILON 93/019 polymer**

Temperature °C	Time days	Tensile strength at yield MPa	Elongation at yield %	Tensile strength at break MPa	Elongation at break %	Tensile modulus <sup>a</sup> GPa	Notched Izod impact strength KJ/m <sup>2</sup>	
85	14	70.8 (0.1)	18.2 (0.1)	29.3 (3)	152 (22)	1.57 (0.07)	14.4 (0.6)	
	28	71.6 (0.3)	17.9 (0.2)	42.3 (8)	160 (68)	1.65 (0.16)	14.9 (0.5)	
	63	73.0 (0.2)	17.2 (0.1)	19.5 (1)	152 (21)	1.70 (0.08)	15.2 (0.6)	
	91	73.3 (0.2)	17.1 (0.5)	26.2 (7)	156 (97)	1.81 (0.04)	15.4 (0.6)	
	119	73.9 (0.1)	17.2 (0.2)	41.5 (5)	154 (46)	1.77 (0.06)	14.7 (0.7)	
	147	74.6 (0.1)	16.8 (0.2)	38.2 (6)	120 (32)	1.70 (0.03)	14.0 (0.5)	
	175	74.8 (0.1)	16.8 (0.2)	48.1 (7)	183 (61)	1.71 (0.01)	14.7 (1.0)	
	230	75.8 (0.1)	17.7 (0.2)	36.9 (2)	144 (27)	1.70 (0.04)	12.6 (0.7)	
	293						11.5 (0.5)	
	349						10.5 (0.4)	
	385		76.5 (0.3)	16.3 (0.05)		86 (51)	1.92 (0.04)	10.4 (1.0)
	100	14	72.0 (0)	19.9 (0.1)	42.4 (4)	149 (25)	1.57 (0.03)	16.6 (0.7)
28		73.0 (0.1)	19.8 (0.3)	41.8 (8)	186 (41)	1.55 (0.11)	17.5 (0.4)	
45		74.1 (0.1)	19.4 (0.1)	49.6 (14)	121 (81)	1.66 (0.04)	16.6 (0.5)	
63		74.9 (0.1)	18.9 (0.1)	22.8 (6)	100 (47)	1.64 (0.12)	16.3 (0.2)	
77		75.3 (0.1)	18.5 (0.1)	22.0 (4)	101 (43)	1.76 (0.06)	15.1 (1.0)	
91		76.2 (0.1)	18.5 (0.2)	21.5 (2)	149 (37)	1.69 (0.09)	15.0 (0.6)	
101		76.6 (0.1)	18.6 (0.1)	49.4 (13)	116 (60)	1.70 (0.03)	13.2 (0.7)	
133		77.7 (0.3)	17.8 (0.3)	55.4 (13)	64 (37)	1.81 (0.11)	11.2 (0.6)	
147							10.4 (0.4)	
161		79.5 (0.1)	16.9 (0.1)	44.0 (0.4)	107 (2)	1.94 (0.04)	9.5 (0.4)	
175							7.0 (2.29)	
189							6.5 (2.3)	
203				74.8 (3.5)	10 (1)	1.82 (0.08)		

<sup>a</sup>= Chord modulus determined between the limits of 0.2 and 0.3% strain  
( )= standard deviation

**Table 5: Effect of thermal ageing on tensile properties and notched Izod impact strength for CARILON 93/019 polymer**

Temperature °C	Time days	Tensile strength at yield MPa	Elongation at yield %	Tensile strength at break MPa	Elongation at break %	Tensile modulus <sup>a</sup> GPa	Notched Izod impact strength KJ/m <sup>2</sup>	
110	3	71.2 (0.1)	20.9 (0.2)	41 (5)	163 (35)	1.42 (0.03)	18.2 (0.5)	
	7	72.2 (0.2)	21.2 (0.1)	48.9 (8)	165 (36)	1.52 (0.05)	18.8 (0.5)	
	14	73.3 (0.1)		44.9 (17)			18.4 (0.5)	
	25	74.5 (0.3)	19.8 (0.3)	49.7 (11)	103 (71)	1.50 (0.05)	15.4 (1.0)	
	28	74.9 (0.2)	20.1 (0.4)	47.8 (13)	97 (44)	1.49 (0.03)	14.9 (0.9)	
	35	75.4 (0.04)	20.6 (0.4)	60.8 (12)	85 (73)	1.50 (0.05)	13.2 (1.0)	
	42	76.4 (0.3)	20.5 (0.3)	56.5 (15)	61 (58)	1.58 (0.04)	13.4 (0.6)	
	49	77.1 (0.1)	20.4 (0.3)	62.3 (14)	31 (21)	1.62 (0.04)	10.9 (1.2)	
	56	77.9 (0.1)	21.1 (0.6)	58.9 (19)	39 (27)	1.56 (0.07)	9.3 (1.9)	
	63	78.5 (0.04)	20.8 (0.6)	62.2 (13)	42 (41)	1.55 (0.03)	8.1 (2.4)	
	70	78.9	20.2	70.1 (9)	19 (14)	1.55 (0.05)	6.6 (2.4)	
	84			48.2 (5)	4.6 (0.6)	1.63 (0.03)	3.7 (1.5)	
	130	3	70.2 (0.2)	24.3 (0.4)	41.0 (12)	146 (41)	1.33 (0.04)	20.6 (0.7)
		7	73.3 (0.2)	23.5 (0.5)	52.5 (9)	115 (66)	1.45 (0.04)	18.2 (0.4)
10		73.6 (0.2)	23.2 (0.7)	54.1 (13)	103 (65)	1.38 (0.04)	16.4 (0.6)	
14		73.7 (4.2)	19.4 (7.0)	51.7 (12)	27 (26)	1.49 (0.03)	14.2 (1.5)	
17				41.6 (4)	4 (1)	1.44 (0.02)	7.9 (3.3)	
21				33.9 (3)	3 (0.5)	1.63 (0.07)	7.0 (1.9)	
24				29.9 (3)	2 (0.3)	1.57 (0.07)	3.6 (1.4)	
28				27.3 (2)	2 (0.6)	1.62 (0.04)	2.6 (0.4)	
31				21.6 (2)	1.5 (0.4)	1.77 (0.08)	2.4 (0.2)	

<sup>a</sup>= Chord modulus determined between the limits of 0.2 and 0.3% strain  
( )= standard deviation

**Table 6:** Effect of thermal ageing on tensile properties and notched Izod impact strength for CARILON 93/019 polymer

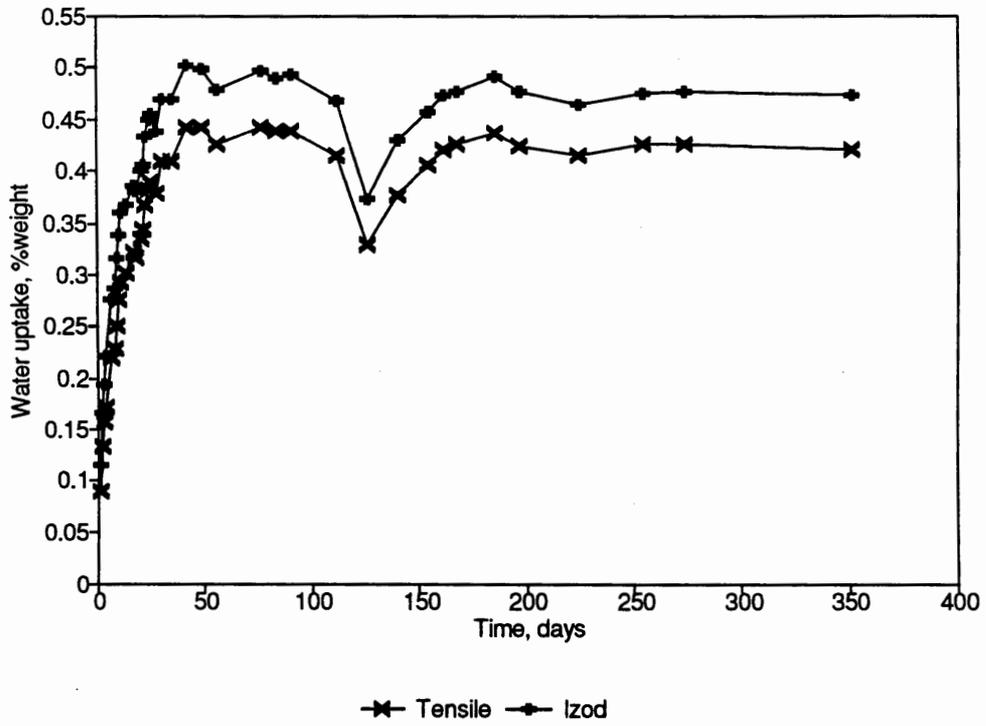
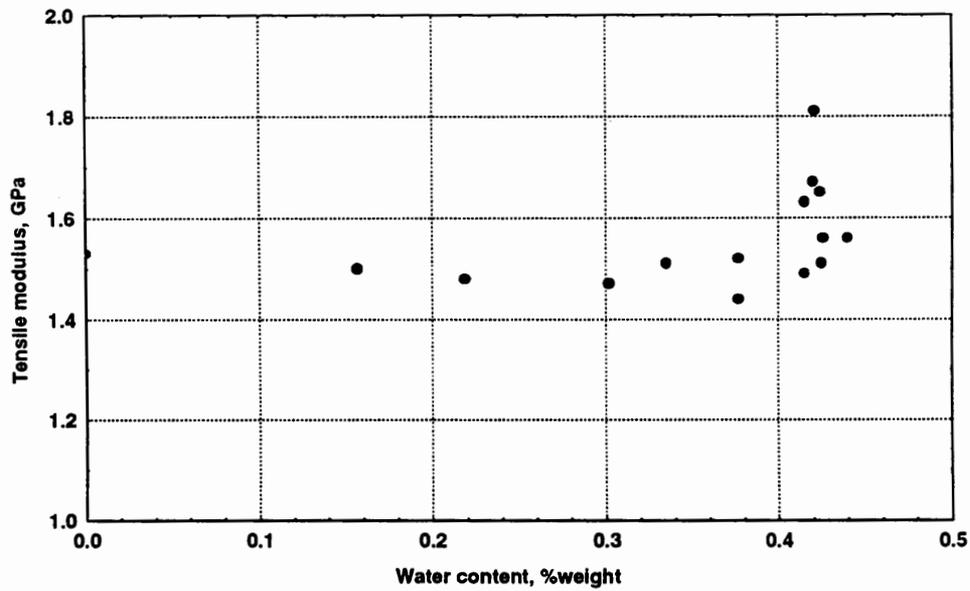
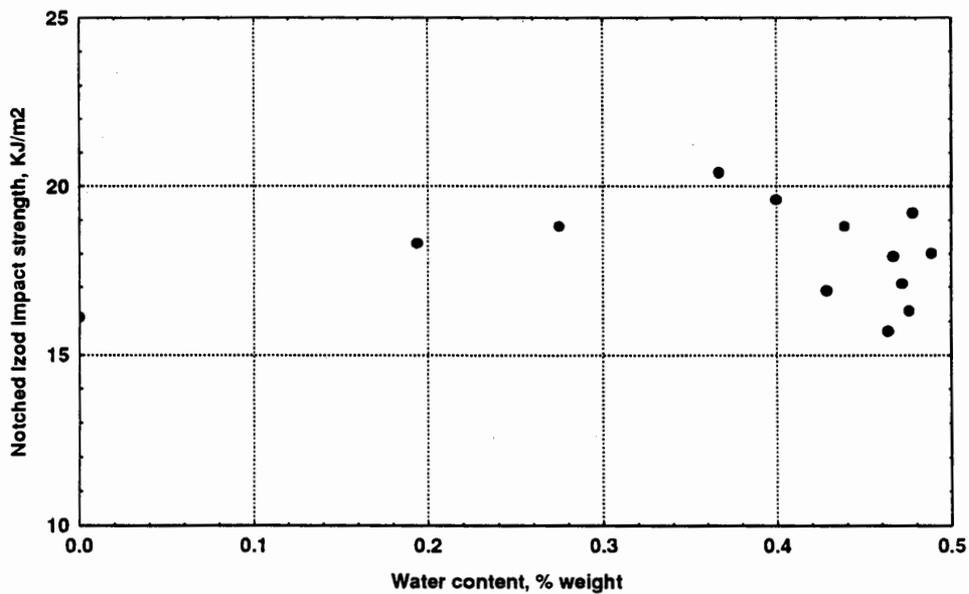


Figure 1: Water uptake of CARILON P1000 batch 93/019 in function of the storage time at 23°C / 50% RH



**Figure 2:** Effect of water content on the tensile modulus in function of storage time at 23°C / 50% RH for CARILON P1000 batch 93/019



**Figure 3:** Effect of water content on the notched Izod impact strength in function of storage time at 23°C / 50% RH for CARILON P1000 batch 93/019

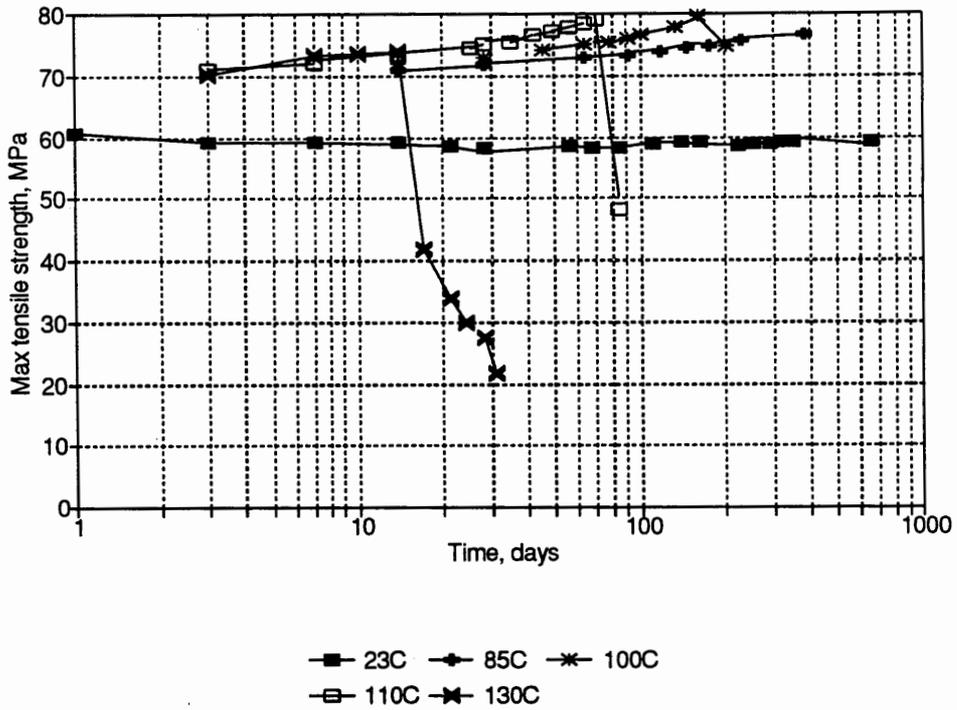


Figure 4: Effect of room temperature storage and thermal ageing on the maximum tensile strength at 23°C for CARILON P1000 batch 93/019

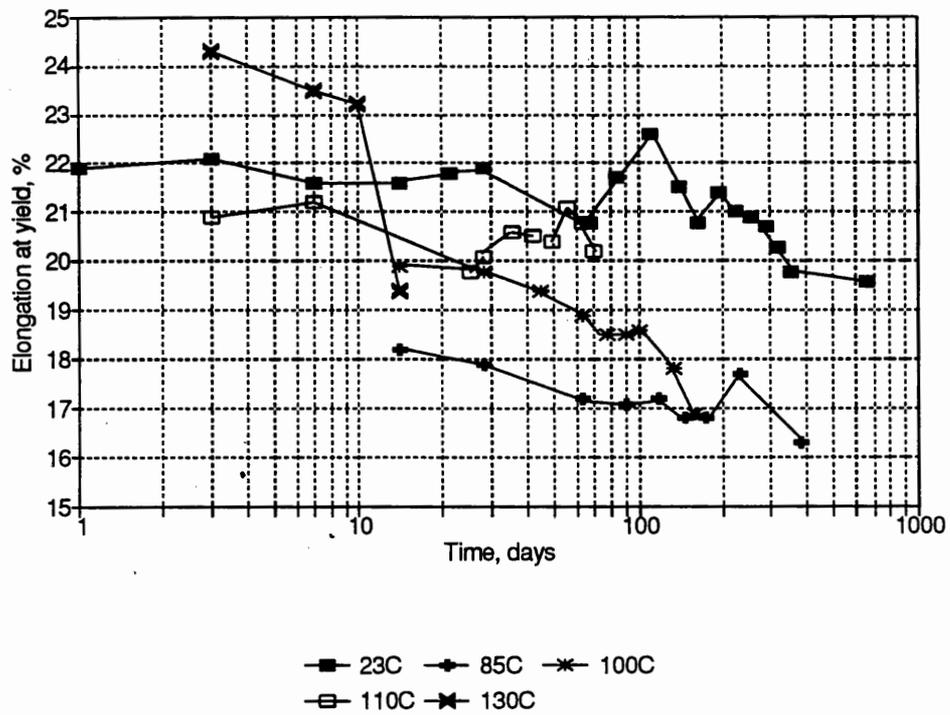


Figure 5: Effect of room temperature storage and thermal ageing on the elongation at yield at 23°C for CARILON P1000 batch 93/019

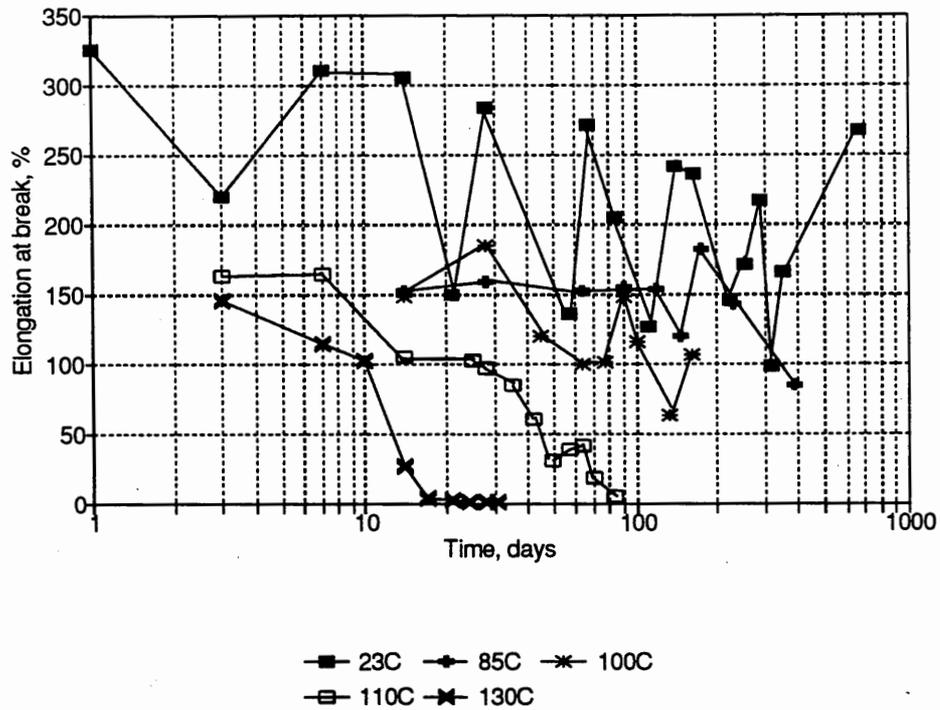


Figure 6: Effect of room temperature storage and thermal ageing on the elongation at break at 23°C for CARILON P1000 batch 93/019

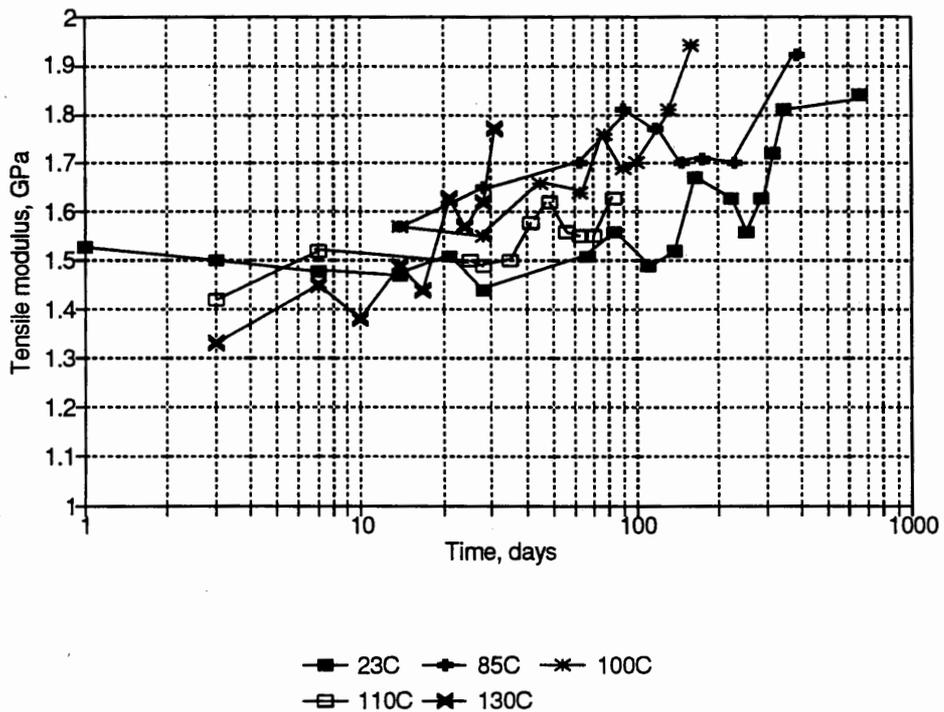


Figure 7: Effect of room temperature storage and thermal ageing on the tensile modulus at 23°C for CARILON P1000 batch 93/019

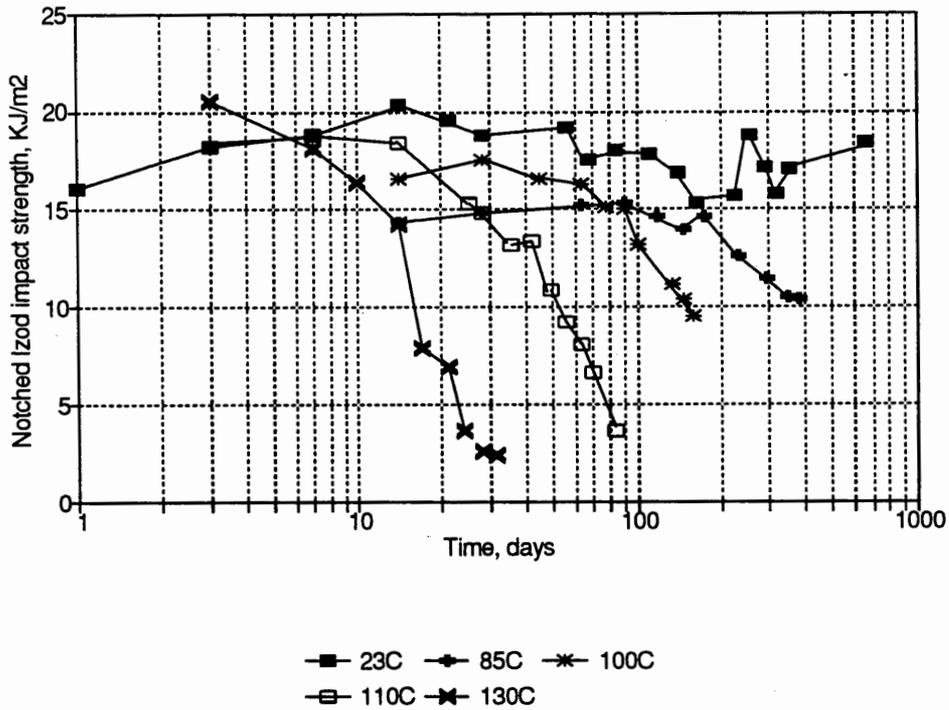


Figure 8: Effect of room temperature storage and thermal ageing on the notched Izod impact strength at 23°C for CARILON P1000 batch 93/019

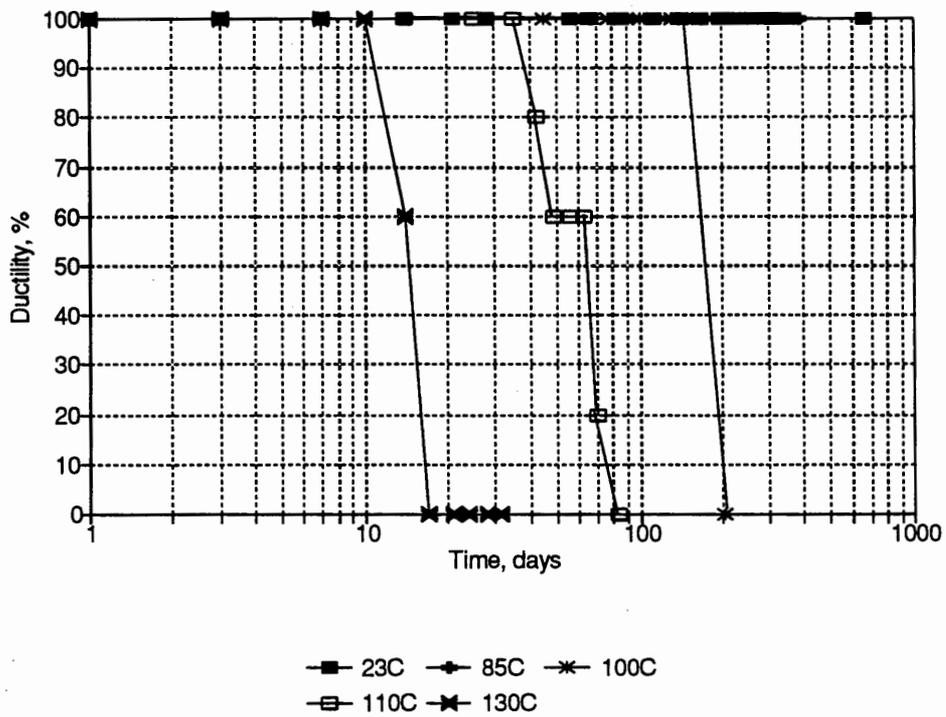
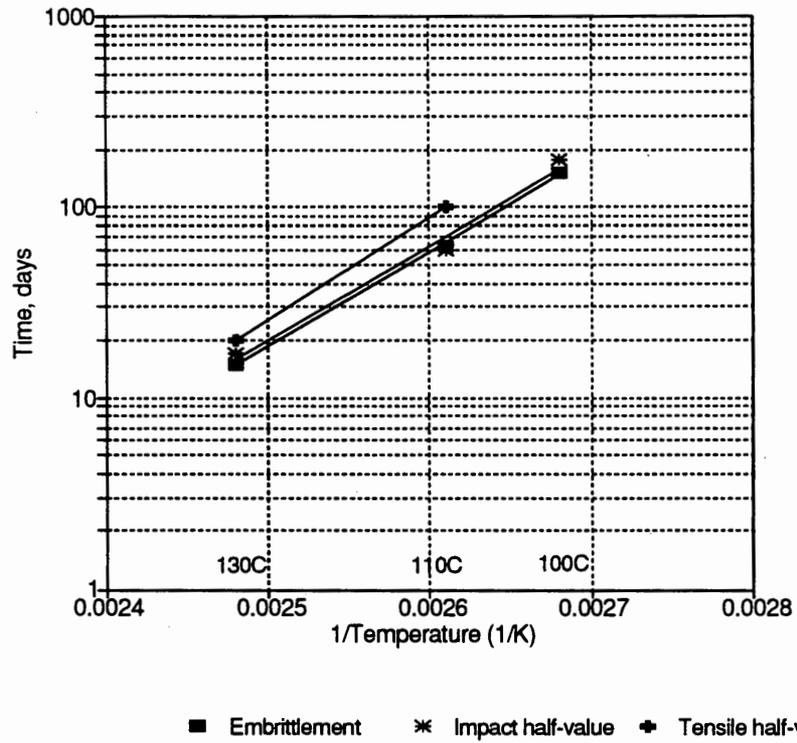


Figure 9: Effect of room temperature storage and thermal ageing on the percentage of samples showing ductility in tensile test



**Figure 10:** Thermal endurance graph based on the onset of embrittlement, notched Izod impact strength half-value and tensile strength half-value criteria for CARILON P1000 batch 93/019

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