Thermal Stability of Butyl/EPDM/Neoprene Based Rubber Compounds

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ABSTRACT: Tyre curing bladders are usually prepared by using butyl/neoprene based compounds. To demonstrate the heat resistance and to extend the service life of a bladder, the mechanical properties of butyl/neoprene/ ethylene–propylene–diene rubber (EPDM) based compounds in different compositions were analyzed and compared. In this study, the effects of partial replacements of IIR with different types of EPDM in different proportions were studied. Rheological and mechanical properties of vulcanizates were measured before and after the thermal ageing. Curing characteristics of unvulcanized compounds and mechanical properties, permanent set, fatigue index, and IRHD hardness of vulcanized compounds were measured. The better results were obtained by using the recipes containing Keltan 27 type EPDM of 10–20 phr. Mechanical properties of recipes decreased due to high EPDM ratio in the recipes studied. The service life of tyre curing bladders can be increased by the replacement of EPDM with butyl rubber in appropriate type and quantity. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 557–563, 2007

Key words: rubber; blends; ageing; mechanical properties; curing bladder

INTRODUCTION

The bladder is an important component in tyre production, because it affects the quality and the cost of tyres. It is used to shape green tyres and as a heat transfer membrane during curing process. Therefore, any increase in the bladder performance can contribute to cost reduction in tyre production.

Curing and shaping of tyres are accomplished by internal and external heat treatment. In curing process, firstly green tyre is placed into curing machine, while the bladder permanently remains in the curing machine. The bladder starts to expand and after a preexpansion period, the mold is closed. High-pressure steam (190–210°C) and/or hot water (190°C) circulate through the bladder to cure the inner component of the tyre. External parts of the tyre, i.e., tread and side wall are cured by mold, which is heated by circulating steam. When the curing time is completed, the bladder is collapsed by applying vacuum to facilitate the easy release of the tyre from the mold.

Butyl rubber (IIR) is widely used in the production of both tyre inner liners and curing bladders due to its good air permeation resistance. However, IIR has a limited resistance to thermal ageing. Therefore, many attempts have been made to overcome this drawback of IIR.¹ In most common bladder recipes, to increase the thermal stability, polychloro-

Journal of Applied Polymer Science, Vol. 103, 557–563 (2007) © 2006 Wiley Periodicals, Inc. prene (neoprene) in 5–10 phr is used as an activator for resin cured butyl rubber. But, in this case, some of the mechanical properties are worsened. Fair heat resistance of IIR can be balanced by blending it with ethylene–propylene–diene rubber (EPDM). Since the permanent set and heat resistance of the EPDM is better than that of butyl rubber, the partial replacement of IIR with EPDM was considered to increase the heat resistance and ageing properties of IIR vulcanizates. It is well known that various types of EPDMs having different structural properties are used in rubber industry. It is obvious that different compound properties can be obtained when different types of EPDM are added to butyl based rubber compounds.

In this study, the effects of partial replacements of IIR with different types of EPDM in different proportions have been studied. Rheological and mechanical properties of vulcanizates were measured before and after the thermal ageing for obtaining formulations with better mechanical properties and good ageing properties; so that the bladders produced with those new compounds can withstand through a greater number of cure cycles. Thus, the overall tyre manufacturing cost can be reduced by the increase in bladder performance.

EXPERIMENTAL

Materials

The butyl rubbers used in this study were Butyl 268 and Butyl 165 from Exxon Chemicals (Buffalo, NY).



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	Principal Prop	erties of EPDM Ty	pes Used in	this Study	
EPDM type	Termonomer type	Mooney viscosity ML 1 + 4 (125°C)	Ethylene content (%)	Termonomer content (%)	Molecular weight distribution
Keltan 27	ENB	68	54	5	Medium
Keltan 708*15Z	ENB	65	67	4.7	Medium
Buna EPG 8450	ENB	76 ± 6	53 ± 4	4.3 ± 0.6	
Buna EPG 6470	ENB	59	71	4.5	

 TABLE I

 Principal Properties of EPDM Types Used in this Study

The ethylene–propylene–diene rubbers (EPDM) were Keltan 27 and Keltan 708*15Z (DSM, Heerlen, The Netherlands), Buna EPG 8450 and Buna EPG 6470 (Bayer, Leverkusen, Germany). The principal characteristics of EPDMs used in this experiment are given in Table I. (Neoprene WRT) was from Dupont (Wilmington, DE). All rubbers studied were investigated as they were provided, because they were within the shelf lives declared by the producers. It is well known that the rubber producers add a small proportion of antioxidant to prevent any shelf degradation. The other compounding ingredients are of commercial grade and customarily being used in tyre industry.

Method

The rubber compounds were prepared in an open tworoll mill. The rheological characteristics of the rubber compounds were determined using a Monsanto MDR 2000E Rheometer. Mooney viscosity and the scorch time were measured by Monsanto MV 2000 and ASTMD 1646. For the testing of the vulcanizates, the compounds were vulcanized in a hydraulic press under a pressure of 13.7 MPa at 178°C for 1 h (predetermined optimum cure time). Mechanical properties of the rubber vulcanizates were measured by using Monsanto T10 tensometer and Monsanto Duratron hardness tester. Dynamic fatigue resistances of the vulcanizates using dumbbell-shape tension specimens were measured by De Mattia Machine in accordance with ASTM D 430. Tests were terminated after the rupture of specimens. An arbitrary fatigue index was defined assuming that the fatigue resistance of the unaged base recipe compound had a value of 100. The ratio of the fatigue resistance of the other samples to the base recipe was defined as "fatigue index." Permanent set values were determined in accordance with ISO 2285 IE at 100°C. The rubber vulcanizates were subjected to thermal ageing in an air-circulated oven at 120°C for different periods. Due to the fact that all vulcanizates can degrade at a temperature of above 120°C, the ageing temperature in air-circulated oven was selected as 120°C after evaluating TGA thermograms of vulcanizates. A Perkin–Elmer TGA 6 thermal analyzer was used.

RESULTS AND DISCUSSION

Effect of butyl grade on mechanical properties

It has been known that butyl 268, having high Mooney grade with high unsaturation, is the most common grade of butyl rubber and still is widely used in bladder applications. Butyl 165 on the other hand, has low Mooney viscosity and medium unsaturation. If better flow properties and/or mechanical properties are required, fairly low unsaturation and low Mooney grades, such as butyl 165 can be blended with butyl 268.

For the aim of evaluating the effects of butyl grade on mechanical properties and deciding on the base recipe to be used in this study, two different bladder compounds were prepared in the beginning. The only difference between the two recipes was in their butyl rubber grades. The recipes and test results of vulcanizates are shown in Table II. As it can be seen from Table II, although the tensile strength, modulus, and elongation at break values for both com-

TABLE II Comparison of Butyl 268 and Butyl 268/165 Blend Rubber Compounds

	Butyl 268	Butyl 268/165 blend
Compound (phr)	-00	
Butyl 268	100	50
Butyl 165	100	50
Neoprene WRT	5	5
Carbon black (N330)	50	50
Castor oil	5	5
ZnO	5	5
Resin	10	10
Cure characteristics		
M_L	2.5	2.3
M_{H}^{-}	7.0	7.8
Mooney viscosity, ML $1 + 4$ (125°C)	79.5	74.0
Scorch time, t_5 (min, @143°C)	59.1	60.1
t_{90} (min)	7.8	8.4
Physico-mechanical properties		
300% Modulus (MPa)	4.1	4.3
Tensile strength (MPa)	12.4	12.7
Elongation at break (%)	736	752
De Mattia fatigue index	69.4	100.0
Hardness (IRHD)	58.4	56.6
Permanent set (%)	21	24

		Buty	1 268			Butyl 268,	/165 blend	
Ageing time (day)	0	1	3	7	0	1	3	7
%300 Modulus (MPa)	4.1	5.2	6.4	7.2	4.3	5.6	6.9	7.3
Tensile strength (MPa)	12.4	12.6	11.0	10.8	12.7	12.7	11.2	11.8
Elongation at break (%)	736	666	518	482	752	669	550	479
Hardness (IRHD)	58.4	63.3	73.5	79.9	56.6	63.0	72.8	77.1
De Mattia fatigue index	69.4	19.3	4.5	0.1	100.0	21.4	4.8	0.1

 TABLE III

 Comparison of Physico-Mechanical Properties of Aged Vulcanizates Prepared with Butyl 268

 and Butyl 268/165 Rubber Blend

pounds are almost the same, the Mooney viscosity and IRHD values of the compound prepared by blending of two butyl grade are lower than that of compound containing only butyl 268. Therefore, the compound with butyl blend exhibits better flow properties during processing. The better advantages of the butyl blend were particularly seen in the fatigue index and in the permanent set values. These two quality parameters have an important effect on the bladder performance. The ageing resistance of the both vulcanizates, in an air-circulated oven at 120°C has also been investigated. The changes in mechanical properties of the compounds with ageing time are given in Table III. As it is clearly seen from the table, the modulus values of both vulcanizates increase with ageing time, while tensile strength and elongation at break values are reduced. The changes in mechanical properties are quite the same for both recipes, but better fatigue resistance and permanent set values in the compounds with butyl blend were observed. After analyzing all the measured properties, the recipe 2 (prepared by blending 2 butyl grades) is chosen as the base bladder compound recipe. Therefore, all the modifications were made on this recipe and the test results pertinent to the modifications were compared with this one.

Thermal stability of butyl/EPDM rubber blends vulcanizates

Although there exist various studies on thermal properties of NR/EPDM blends and curing characteristic and mechanical properties of NR/Chloroprene in literature, it has been reported very limited study on thermal stability of butyl/EPDM rubber blend vulcanizates.²⁻⁵ It is well known that various types of EPDMs having different structural properties are available and being used in rubber industry. Using EPDM containing low ethylidene norbornene (ENB) has some advantages for obtaining good mechanical properties and acceptable permanent set values.^o Therefore, EPDM types containing ENB as termonomer are preferred in this study for partial replacement of butyl in new recipes. Viscosities of EPDMs were varied in a wide range to investigate the effects of viscosity on rheological properties. Selected EPDM rubbers in various proportions, i.e., 10, 15, 20, 25, and 30 phr were replaced with butyl rubber in the base bladder recipe. Other components of the recipes remained unchanged. The compositions of the blends used in this study are shown in Table IV.

The rheological and mechanical properties of compounds with butyl/EPDM blends in different proportions and different types of EPDM and the comparison of the same characteristics with the base recipe are given in Tables V–VIII. As it can be seen from the tables, compound viscosities increase drastically with EPDM blending with Buna EPG 8450 and Keltan 27 and increase moderately with Keltan 708*15Z and with Buna EPG 6470. This is in conformity with Mooney viscosities of EPDMs indicated in Table I. The compound viscosities increase also with the increase in the quantity of EPDM. Scorch values, on the other hand, were varied depending on types of EPDM. A decreases in scorch values with Keltan 708*15Z and an increase with Keltan 27 were ob-

 TABLE IV

 Compound Recipes Prepared by Replacing Butyl with EPDM (Remaining Components as per the Base Recipe)

-		-	-		•	-	0	•					-	-		-			-	
		Kelt	an 27	(phr)		ŀ	Keltan	708*15	Z (ph	r)	E	Buna E	PG 84	50 (ph	r)	E	Buna E	PG 64	70 (ph	r)
	KE1	KE	KE3	KE4	KE5	KZ1	KZ2	KZ3	KZ4	KZ5	BA1	BA2	BA3	BA4	BA5	BU1	BU2	BU3	BU4	BU5
Butyl 268	45	42.5	40	37.5	35	45	42.5	40	37.5	35	45	42.5	40	37.5	35	45	42.5	40	37.5	35
Butyl 165	45	42.5	40	37.5	35	45	42.5	40	37.5	35	45	42.5	40	37.5	35	45	42.5	40	37.5	35
Keltan 27	10	15	20	25	30															
Keltan 708*15Z						10	15	20	25	30										
Buna EPG 8450											10	15	20	25	30					
Buna EPG 6470																10	15	20	25	30

	Base recipe	KE1	KE2	KE3	KE4	KE5
Cure characteristics						
M_L	2.3	2.0	2.1	2.1	2.1	2.1
M_H	7.8	6.6	7.3	7.5	7.7	7.5
Mooney viscosity, ML $1 + 4$ (125°C)	74	77	81	85	85	87
Scorch time, t_5 (min, @143°C)	60.1	91.7	76.3	83.0	69.0	71.2
t_{90} (min)	8.4	8.7	8.6	8.7	8.6	8.7
Physico-mechanical properties						
300% Modulus (MPa)	4.3	4.7	6.0	6.1	6.2	6.1
Tensile strength (MPa)	12.7	12.7	13.5	13.3	13.0	12.8
Elongation at break (%)	752	707	695	653	635	606
De Mattia fatigue index	100.0	40.9	28.2	25.1	6.9	3.4
Hardness (IRHD)	56.6	61.6	62.5	64.3	61.2	62.5
Permanent set (%)	24	22	22	22	24	28

TABLE V Rheological and Mechanical Properties of Butyl/Keltan 27 Blend Rubber Compounds and Vulcanizates

TABLE VI
Rheological and Mechanical Properties of Butyl/Keltan 708*15Z Blend Rubber
Compounds and Vulcanizates

	Base recipe	KZ1	KZ2	KZ3	KZ4	KZ5
Cure characteristics						
M_L	2.3	2.0	1.8	2.2	1.9	2.0
M_H	7.8	7.7	7.2	9.1	8.5	8.9
Mooney viscosity, ML $1 + 4$ (125°C)	74.0	70.7	77.3	69.6	70.1	77.1
Scorch time, t_5 (min, @143°C)	60.1	42.4	44.8	36.3	40.9	40.0
t ₉₀ (min)	8.4	8.3	8.5	8.3	8.4	8.4
Physico-mechanical properties						
300% Modulus (MPa)	4.3	6.0	6.0	6.2	7.1	7.2
Tensile strength (MPa)	12.7	12.0	12.1	12.1	10.7	11.5
Elongation at break (%)	752	598	595	590	465	490
De Mattia fatigue index	100.0	38.6	27.8	14.9	2.8	0.1
Hardness (IRHD)	56.6	62.2	63.6	62.1	64.9	67.7
Permanent set (%)	24	24	30	30	30	31

TABLE VII

Rheological and Mechanical Properties of Butyl/Buna EPG 8450 Blend Rubber Compounds and Vulcanizates

	Base recipe	BA1	BA2	BA3	BA4	BA5
Cure characteristics						
M_L	2.3	2.1	1.9	2.2	2.3	2.3
M_H	7.8	8.2	6.5	8.9	9.6	9.1
Mooney viscosity, ML $1 + 4$ (125°C)	74.0	81.3	80.0	84.3	94.4	92.6
Scorch time, t_5 (min, @143°C)	60.1	51.6	78.0	46.9	43.9	58.6
t_{90} (min)	8.4	8.3	8.7	8.4	8.5	8.5
Physico-mechanical properties						
300% Modulus (MPa)	4.3	4.9	4.6	6.2	6.7	6.2
Tensile strength (MPa)	12.7	12.7	13.3	12.7	13.2	13.8
Elongation at break (%)	752	665	700	600	595	600
De Mattia fatigue index	100.0	31.1	21.7	7.3	3.0	0.1
Hardness (IRHD)	56.6	64.4	61.1	63.8	64.6	64.5
Permanent set (%)	24	22	28	28	23	24

served while the scorch values almost remained unchanged with Buna EPG 8450 and Buna EPG 6470. As it can be seen in Table I, changes in scorch values with EPDM blending were found to be in parallel with the termonomer contents of used EPDMs. This behavior can be explained with the low termonomer contents of Buna EPDMs with respect to the Keltan EPDMs.

Changes in physicomechanical properties with increasing EPDM quantity were also investigated. The 300% modulus and IRHD hardness values were increased with increasing EPDM quantity in all types

	1					
	Base recipe	BU1	BU2	BU3	BU4	BU5
Cure characteristics						
M_L	2.3	2.0	1.9	2.0	1.9	1.9
M_H	7.8	6.9	6.7	7.2	7.8	7.9
Mooney viscosity, ML $1 + 4$ (125°C)	74.0	72.9	70.5	79.4	78.1	78.8
Scorch time, t_5 (min, @143°C)	60.1	60.4	68.2	59.7	53.8	58.2
t ₉₀ (min)	8.4	8.6	8.8	8.7	8.6	8.7
Physico-mechanical properties						
300% Modulus (MPa)	4.3	5.8	6.0	7.2	7.9	8.2
Tensile strength (MPa)	12.7	13.2	12.7	11.9	12.9	13.5
Elongation at break (%)	752	671	645	530	532	520
De Mattia fatigue index	100.0	38.6	19.4	7.4	2.8	0.1
Hardness (IRHD)	56.6	61.3	60.7	64.8	65.6	69.5
Permanent set (%)	24	33	32	36	39	39

TABLE VIII Rheological and Mechanical Properties of Butyl/Buna EPG 6470 Blend Rubber Compounds and Vulcanizates

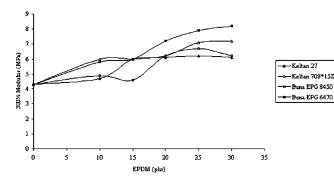


Figure 1 Effect of EPDM type and quantity on 300% modulus of butyl/EPDM rubber blend vulcanizates.

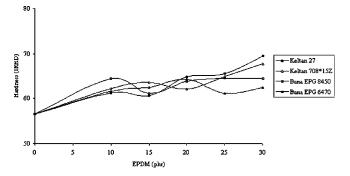


Figure 2 Effect of EPDM type and quantity on IRHD hardness of butyl/EPDM rubber blend vulcanizates.

of EPDMs as can be seen in Figures 1 and 2. Tensile strength values, on the other hand, remained almost unchanged (Fig. 3) while elongations at break were reduced. Decrease in elongation values at break are quite slow with Keltan 27 and are clear evident with Keltan 708*15Z. The Buna EPDMs remained between these values.

The permanent set value has a great importance for a bladder compound, because the bladders made of the compounds with higher permanent set, undergo dimensional changes during repetitive vulcanization cycles. The expanding of the bladder during

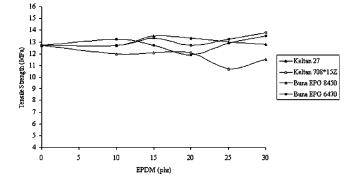


Figure 3 Effect of EPDM type and quantity on tensile strength of butyl/EPDM rubber blend vulcanizates.

its life can provoke tyre scrap due to folding of the bladder which is a principal quality problem in curing department. Therefore, the permanent set values of compounds were also investigated. The permanent sets were slightly decreased with Keltan 27 up to 20 phr and then were increased with the increase of the quantity of EPDM as seen in Table V. The permanent set values were increased considerably for Buna EPG 6470 and Keltan 708*15Z and were increased slightly for Buna EPG 8450.

Fatigue resistance of compound reflects directly the bladder performance, i.e., bladder life. The fatigue indexes decrease with increasing EPDM contents in all unaged compounds. This decrease is slower for Keltan 27 and faster for Buna EPG 6470.

The ageing resistance of butyl/EPDM blend vulcanizant are of an interesting nature. The samples were aged in an air-circulated oven at 120°C and then mechanical properties were measured. The physicomechanical measurements on aged samples are given in Tables IX–XII. As it is seen from those tables, thermal ageing of butyl/EPDM results in increases 300% modulus and IRHD hardness values, but decreases in elongation at breaks, tensile strength, and fatigue resistance values. These changes are more evident and higher in case of Buna EPG 8450 and Buna EPG

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	KE5		9.3 13.6 457 69.5 2.2			KZ5	1	9.2 12.0	434	0.1.0		B/		8.6 14.1 478	$69.1 \\ 0.1$
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		~	$10.2 \\ 11.5 \\ 381 \\ 82.1 \\ 1.0 \\ 1.0$				7	$9.7 \\ 10.4$	364 22 -	6.28 0.6			4	9.8 11.9 374	81.3 0.1
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eing	KE4		8.5 8.5 500 5.2 5.2		Ageing	KZ4	1	6.6 10.1	434 20	69.6 1.4	Ageing	BA4		8.3 13.1 477	70.1 1.5
TABLE IX Physico-Mechanical Properties of Butyl/Keltan 27 Vulcanizates After Ageing		0	6.2 13.0 635 61.2 6.9		TABLE X al Properties of Butyl/Keltan 708*15Z Vulcanizates After Ageing		0	$7.1 \\ 10.7$			TABLE XI al Properties of Butyl/Buna EPG 8450 Vulcanizates After Ageing		0	6.7 13.2 595	64.6 3.0
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al Prop	KE2		7.5 11.8 522 4 66.5 11.9		Proper	KZ2	1		519 519 5		Proper	BA2		7.0 14.3 607 4	
echanic		0	6.0 13.5 695 5 62.5 28.2 28.2		anical		0	6.0 12.1		63.6 27.8			0	4.6 13.3 700 6	
sico-Me		 ~	8.3 11.5 465 81.0 5.9 5.9 2		Physico-Mechanic		7	8.4 11.7		2.5	Physico-Mechanic		 ~		79.1 0.1
Phys		33	8.1 12.5 1 493 46 71.4 8 13.4		Physico		3	6.6 12.7 1		68.3 5.2	Physice		3	9.1	66
	KE1					KZ1				66.0 6		BA1		6.4 7. 14.0 12. 637 484	9
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		0	4.7 12.7) 707 61.6 x 40.9				0	6.0 12.0	L)	x 38.6			0	66 7	
		Ageing time (day)	%300 Modulus (MPa) Tensile strength (MPa) Elongation at break (%) Hardness (IRHD) De Mattia fatigue index				Ageing time (day)	%300 Modulus (MPa) Tensile strength (MPa)	Elongation at break (%)	Hardness (IKHU) De Mattia fatigue index			Ageing time (day)	%300 Modulus (MPa) Tensile strength (MPa) Elongation at break (%)	Hardness (IRHD) De Mattia fatigue index

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6470, but are lower in case of Keltan blends. It has been observed that better thermal resistance values were obtained with the butyl/Keltan blends up to 20 phr of EPDM with respect to the base recipe. The higher EPDM quantity above 20 phr starts to affect the thermal resistance of vulcanizates adversely.

CONCLUSIONS

This study emphasizes the effects of EPDM blending with butyl rubbers on mechanical properties, permanent set, fatigue index, and IRHD hardness of vulcanized compounds. Aged and unaged samples were used to examine how the compound recipes of bladder affect the bladder performance due to the heat resistance of the compounds. Better results were obtained by using the recipes containing Keltan 27 type EPDM of 10–20 phr. Mechanical properties of recipes were decreased due to high EPDM ratio in recipes tested.

It is well known that fatigue resistance of compound reflects directly the bladder performance, i.e., bladder life. The fatigue indexes decreased with increasing EPDM contents in all compounds. The loss of De Mattia fatigue index was slower for Keltan 27 recipe than the other recipes studied. It was shown that the type and quantity of the EPDM used affect the mechanical and thermal stability of bladder compounds.

In conclusion, the service life of tyre curing bladders can be increased by the replacement of butyl rubber in the bladder compound with EPDM in appropriate type and quantity.

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	BU1			BU.	5			BU3	3			BU2 BU3 BU3 BU4	4	t		BU5	15	t
3			0	-			0	-			0	-	n		0	-	n	~
5.8 7.8 8.5 9.3 6.0	5 9.3		6.0	7.6	8.3	9.5	7.2	9.7	10.5	11.7	7.9	9.1	9.8	10.8	8.2	10.9	12.1	
13.2 13.1 13.3 12.1 12.7	3 12.1		12.7	12.9	11.4	11.8	11.9	13.6	13.1	12.4	12.9	13.2	11.6	11.8	13.5	14.2	13.1	12.7
533 492 411 6		Q	645	539	436	414	530	461	414	340	532	475	386	358	520	425	353	299
67.9 70.4 79.9 60.7			60.7	67.5	71.0	81.8	64.8	71.4	73.0	82.6	65.6	70.5	73.5	81.0	69.5	71.6	76.9	84.9
17.8 5.9 0.1 1		Η	9.4	8.9	4.5	0.1	7.4	4.5	3.0	0.1	2.8	2.7	1.4	0.1	0.1	0.1	0.1	0.1

TABLE XII