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Unternehmensgruppe Hoffmann

Improving the processing properties of SBR-silica compounds by the addition of Neuburg Siliceous Earth

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Summary

Although increasing the diethylene glycol content improves the processing properties of the compound, mechanical values are negatively influenced.

The addition of an aid can also improve processing properties, but tensile strength and tear resistance are weakened.

However two other options exist to improve processing properties without significantly affecting mechanical properties:

Precipitated silica can be partially replaced by Sillitin Z 86 where compression set, very low Mooney viscosity rate and high vulcanization properties with long scorch time are required.

Where high tensile strength, tear resistance, high modulus and rebound elasticity are a priority, precipitated silica can be partially replaced with Sillitin Z 86, at the same time reducing the amount of DEG by using less softener.

CONTENT

1 Introduction

The use of precipitated silica as a reinforcing filler in rubber compounds results in improved mechanical properties.

But while good mechanical values can be achieved using this method, compounds containing such fillers can be very difficult to process. This negative and extremely disruptive effect can be found where medium to high hardness is processed, as filler dosage is at its highest here.

With inactive fillers the reverse is often true. Here processing values are frequently good at the cost of only moderate mechanical values.

Active non-black fillers provide three means of achieving good processing values:

- Use of filler (de)-activators
- Addition of processing aids
- Combination of active and inactive fillers

At the same time good mechanical properties are not affected.

The objective of this study was to find a solution to this problem using a non-black SBR compound as a sample.

2 Study Plan

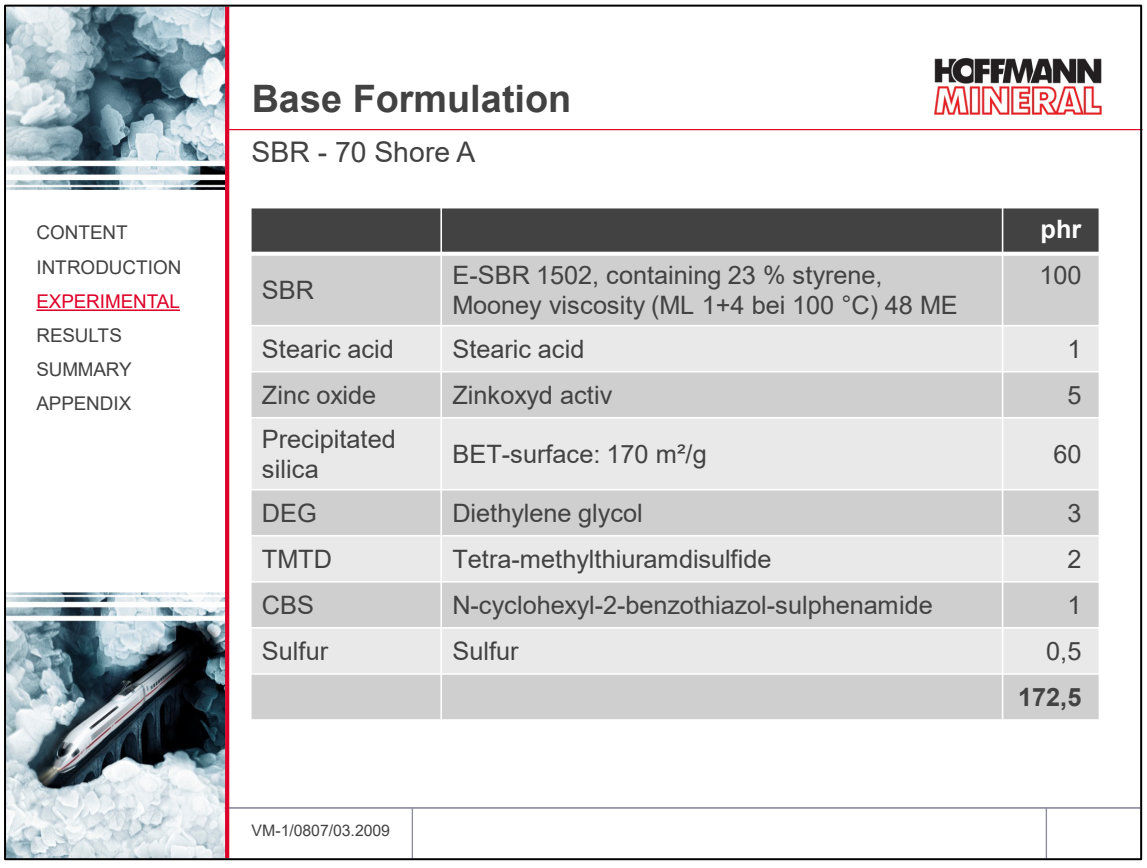
2.1 Objectives

Processing characteristics can be determined directly from the Mooney viscosity of the compound. The objective of the study, therefore, was to considerably reduce Mooney viscosity, at the same time maintaining the compound's mechanical properties.

2.2 Base Formulation

The base formulation comprised a sulfur cured SBR compound filled with precipitated silica and having a hardness of 70 Shore A. A compound containing 60 phr precipitated silica (without DEG) was found to have extremely high Mooney viscosity and unsatisfactory vulcanization characteristics, making the production of reproducible vulcanizates not possible. A processable compound could only be obtained by using a filler desactivator (5 % DEG related to amount of precipitated silica[1]).

The base formulation was optimized as follows:



		phr
SBR	E-SBR 1502, containing 23 % styrene, Mooney viscosity (ML 1+4 bei 100 °C) 48 ME	100
Stearic acid	Stearic acid	1
Zinc oxide	Zinkoxyd activ	5
Precipitated silica	BET-surface: 170 m²/g	60
DEG	Diethylene glycol	3
TMTD	Tetra-methylthiuramdisulfide	2
CBS	N-cyclohexyl-2-benzothiazol-sulphenamide	1
Sulfur	Sulfur	0,5
		172,5

[1] This ratio applies throughout this report

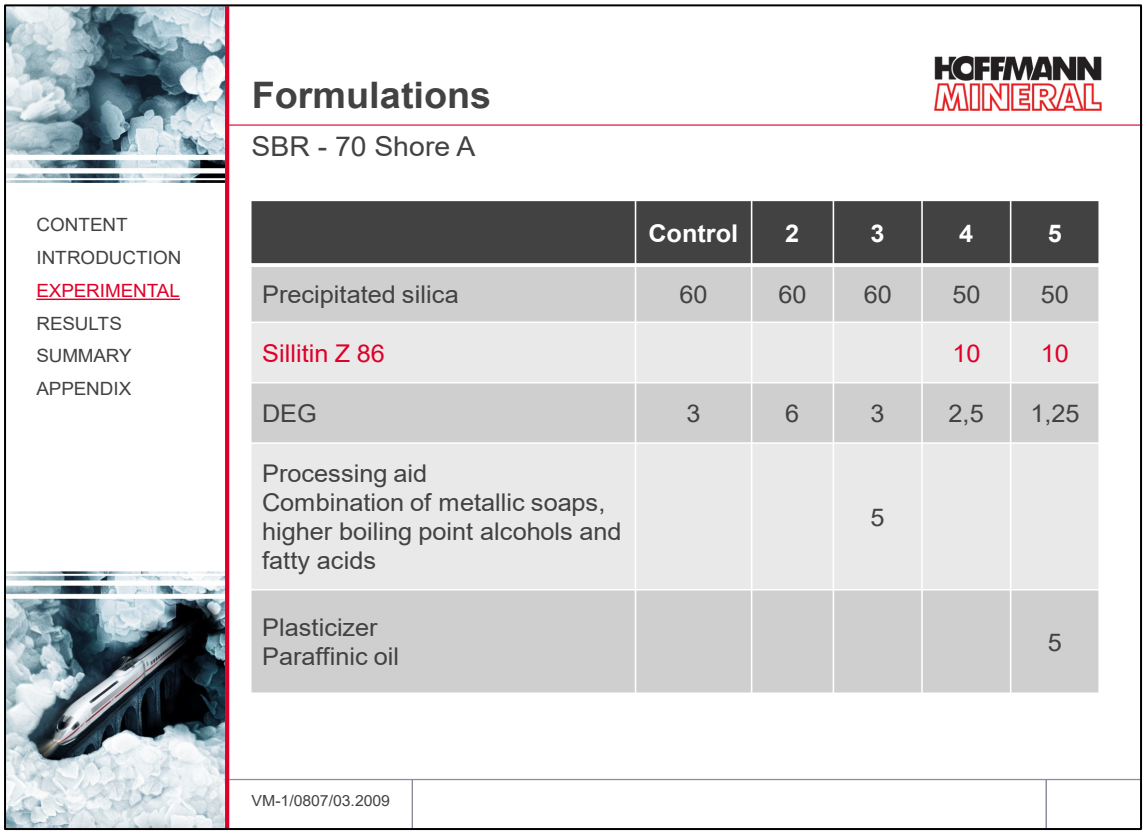
2.3 Method

The starting point was the base formulation.

The following changes were made to the formulation:

- Increased amount of diethylene glycol (No. 2)
- Use of processing aid at a constant level of diethylene glycol (No. 3)
- Partial replacement of precipitated silica by Sillitin Z 86 while maintaining a constant ratio of precipitated silica to diethylene glycol No. 4)
- Reduction of diethylene glycol and addition of softener while maintaining the same amount of Sillitin Z 86 (No. 5).

This results in the following formulations:



	Control	2	3	4	5
Precipitated silica	60	60	60	50	50
Sillitin Z 86				10	10
DEG	3	6	3	2,5	1,25
Processing aid Combination of metallic soaps, higher boiling point alcohols and fatty acids			5		
Plasticizer Paraffinic oil					5

Raw materials used:

SBR:	E-SBR 1502, containing 23% styrol, Mooney viscosity (ML 1+4 at 100 °C) 48
Zinc oxide:	Zinkoxid aktiv Precipitated silica
BET Surface:	170 m^2/g
Neuburg Siliceous Earth:	Sillitin Z 86
Softener:	Paraffin oil
DEG:	Diethylene glycol
Processing aid:	Combination of metallic soaps, higher boiling point alcohols and fatty acids
TMTD:	Tetra-methylthiuramdisulfide
CBS:	N-cyclohexyl-2-benzothiazolsulphenamide

3 Preparation of the Compound

The compound was made up using a laboratory roller (Schwabenthan Polymix 150 L). The rubber was added to the roller at 50° C, followed by the remaining ingredients in the order given in the formulation, at the same time keeping the roller at a constant temperature. Mixing time was around 15 minutes on average.

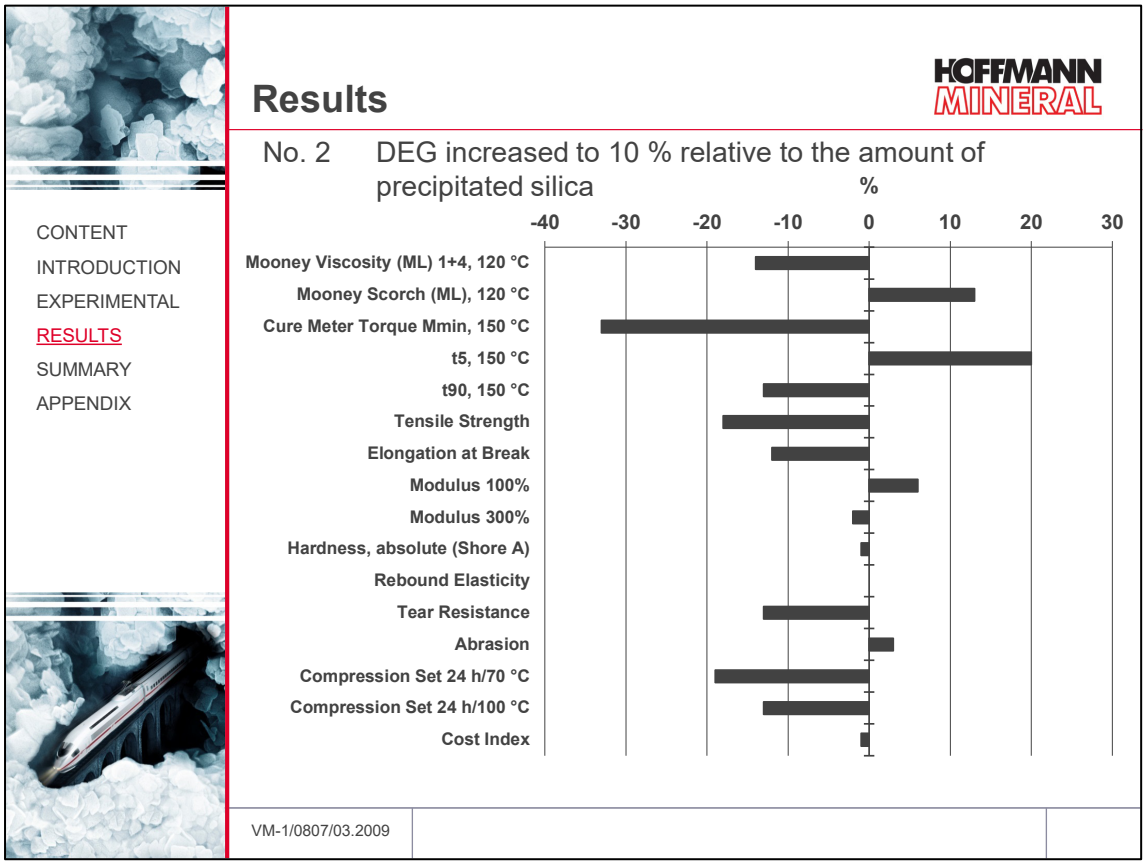
4 Results

The values obtained are shown in the annex.

The cost index relates to prices in Germany in 1996.
The base formulation has an index value of 100.

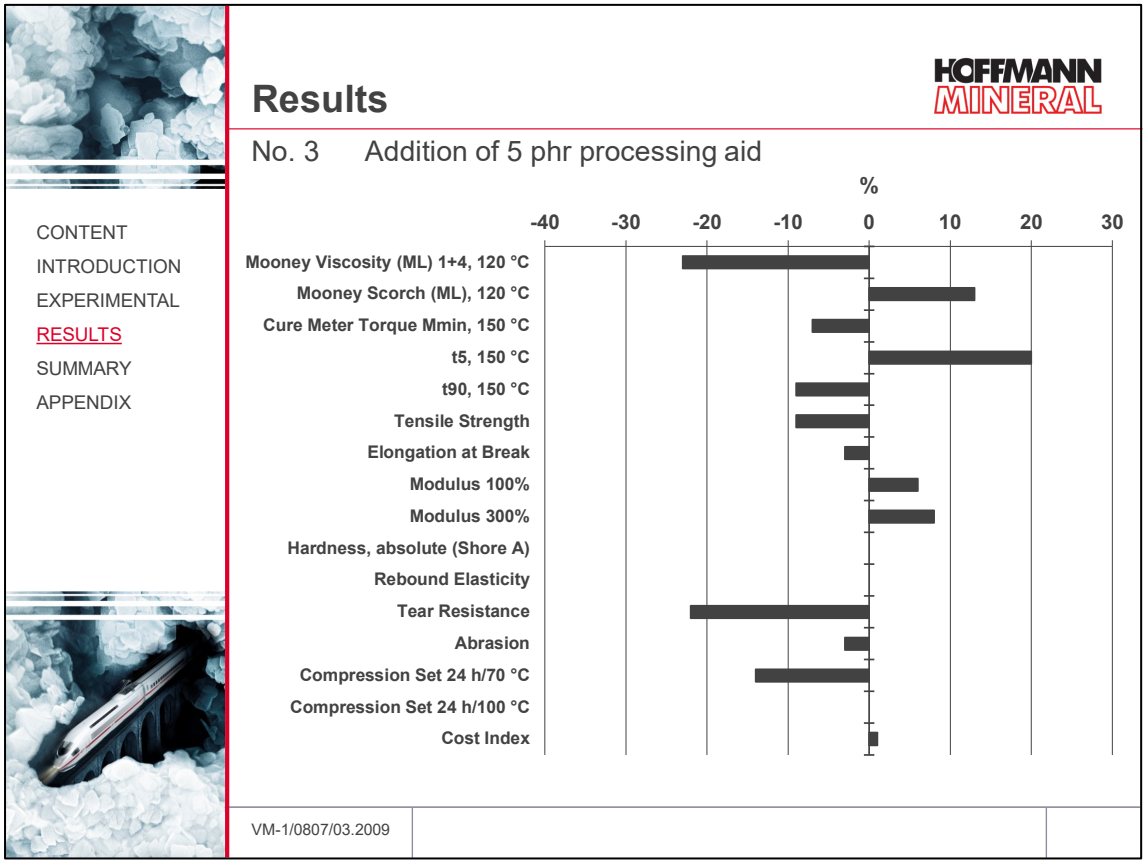
4.1 Increasing DEG

The amount of DEG was doubled to 10 % relative to the amount of precipitated silica (Formulation No. 2).



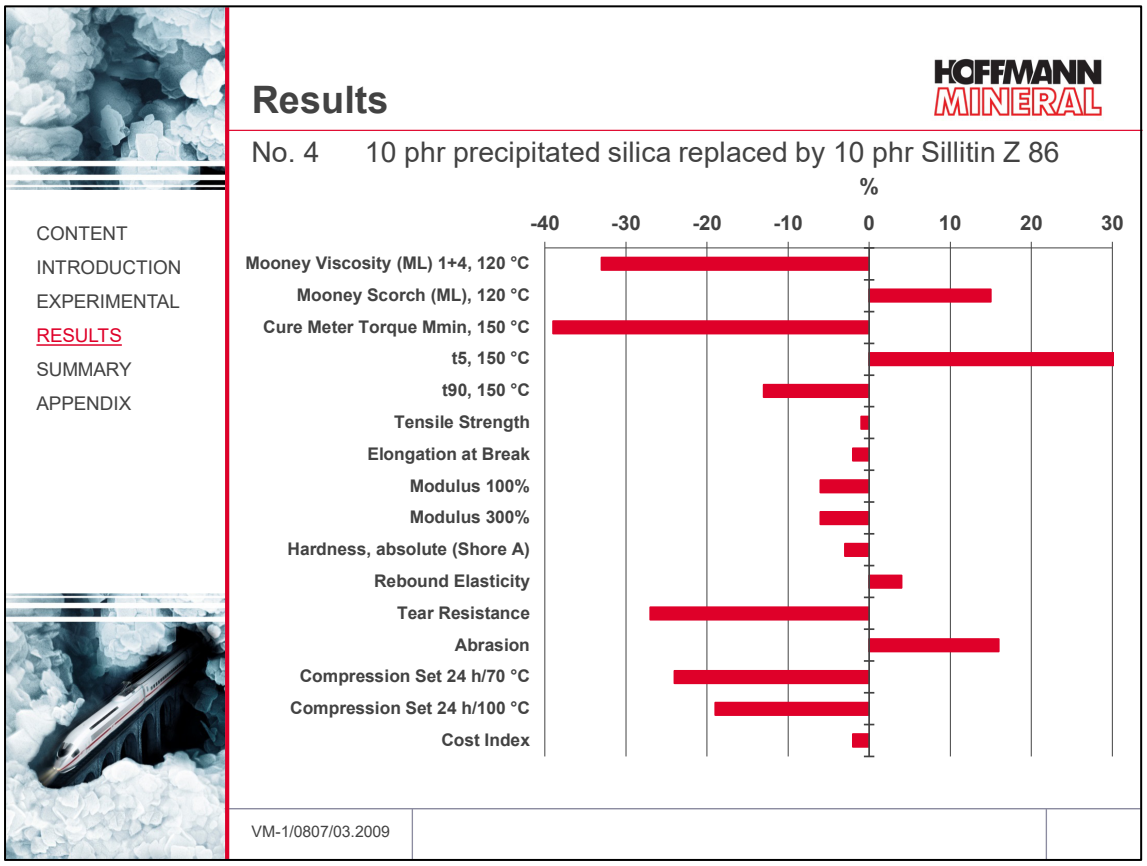
4.2 Use of Processing Aid

5 phr processing aid was used (Formulation No. 3).



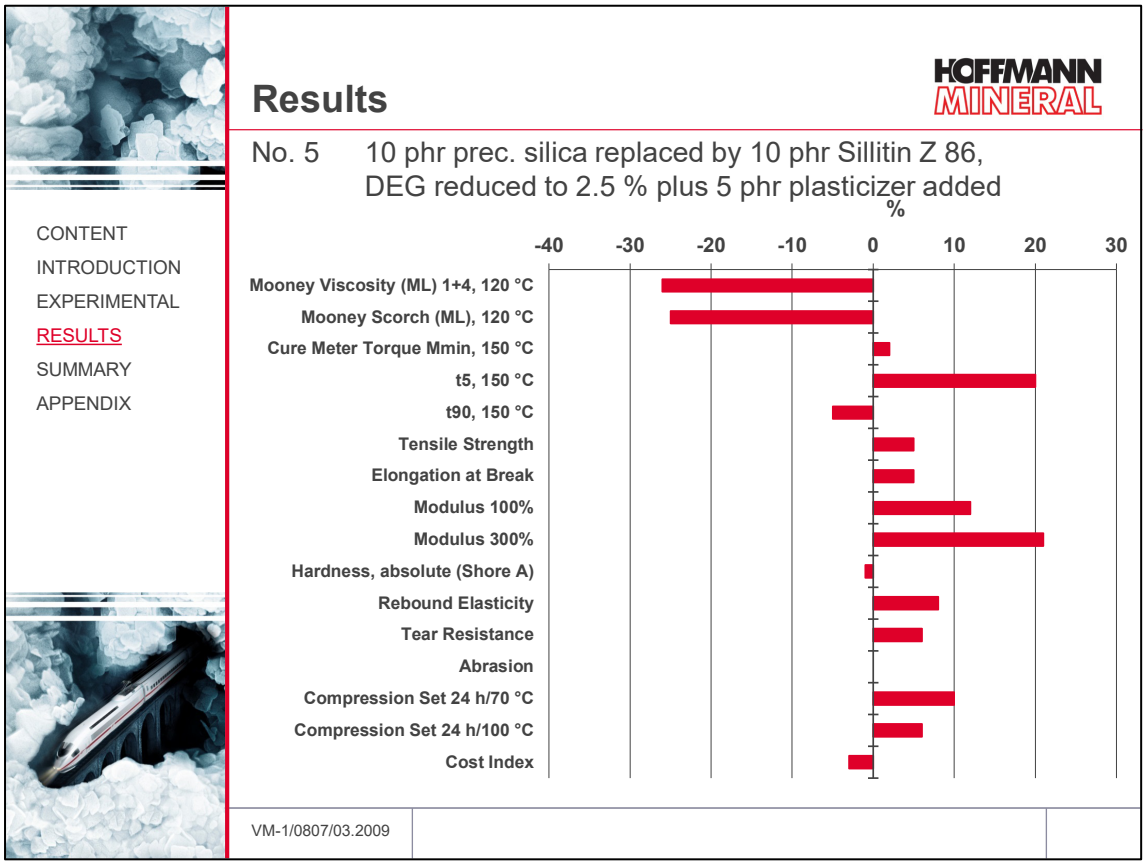
4.3 Partial Replacement of Precipitated Silica with Sillitin Z 86

10 phr precipitated silica was replaced with 10 phr Sillitin Z 86 at a constant ratio of precipitated silica to DEG (Formulation No. 4).



4.4 Partial Replacement of Precipitated Silica with Sillitin Z 86 andReduction of DEG plus Addition of Softener

The precipitated silica/DEG ratio was halfed compared to Formulation No. 4, 5 phr softener was added (Formulation No. 5).



5 Appendix

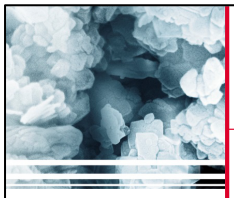


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Table of Test Results

HOFFMANN MINERAL

Properties

				DEG in-creased	+ Pro-cessing aid	Sillitin Z 86	Sillitin Z 86 - DEG + plasti-cizer
			Control	2	3	4	5
Mooney viscosity (ML) 1+4, 120 °C	DIN 53523, T3	ME	107	92	82	72	79
Mooney scorch (ML), 120 °C	DIN 53523, T4	min	10,6	12,0	12,0	12,2	8,0
Cure meter Göttfert Elastograph (0,2°/150 °C)							
Torque M_{min}	DIN 53529, A3	Nm	0,46	0,31	0,43	0,28	0,47
Viscosity t_5	DIN 53529, A3	min	0,5	0,6	0,6	0,9	0,6
Viscosity t_{90}	DIN 53529, A3	min	10,4	9,0	9,5	9,1	9,9
Conversion time T_{90} + 10 %		min	11,5	9,9	10,5	10,0	10,9

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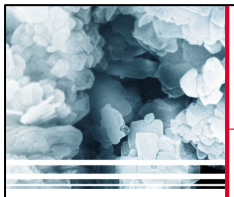

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			Control	2	3	4	5
Tensile Strength	DIN 53504, S2	MPa	16,0	13,2	14,5	15,8	16,8
Elongation at break	DIN 53504, S2	%	590	520	570	580	620
Modulus 100 %	DIN 53504, S2	MPa	1,7	1,8	1,8	1,6	1,9
Modulus 100 %	DIN 53504, S2	MPa	4,8	4,7	5,2	4,5	5,8
Hardness	DIN 53505	Shore A	70	69	70	67	69
Rebound	DIN 53512	%	48	48	48	50	52
Tear Resistance	DIN ISO 34-1	N/mm	14,4	12,6	11,3	10,5	15,2
Abrasion	DIN ISO 4649	mm³	190	195	185	220	190
Compression set 25 h/70 °C, 25 % Deflection	DIN ISO 815	%	21	17	18	16	23
Compression set 25 h/100 °C, 25 % Deflection	DIN ISO 815	%	32	28	32	26	34
Volume cost index*			100	99	101	98	97

*The base formulation has an index value of 100 and is valid for Germany.

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