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TEST REPORT

"Mixing and physical tests of 3 MgO grades
BIIR recipe"

DIK order no.: G20L0093
Customer order no.: Contract vom 27.01.2020

Customer:

LLC Vyazma-Brucite

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Testing site: Material testing
Person in charge: J. Heier

1 Specimen description

Table 1: Submitted sample

Specimen designation Customer	Specimen designation DIK	Description	Date received
MgO Competitor 1	G20L0093/1	Synthetic MgO	28 Jan 2020
MgO Competitor 2	G20L0093/2	Synthetic MgO	28 Jan 2020
MagPro 150	G20L0093/3	Natural MgO	30 Jan 2020

2 Objectives

This report is consisting of the BIIR trials only. So mixing and physical tests of 3 MgO grades in **halo butyl rubber (BIIR)** was to be performed. The compounds were to be tested in unvulcanized and vulcanized state.

3 Test methodology

- Mixing of an customer halo-butyl rubber recipe
- Measurement of Mooney viscosity ML 1+4 100 °C,
In accordance with DIN ISO 289-1:2018-12
- Investigation of Mooney scorch behavior tested at 125 °C time to 5 pts and 10 pts rise, in accordance with DIN ISO 289-2:2018-12
- Measurement of rheometer curves at 160 °C 60 min,
In accordance with DIN 53529-3:1983-06
- Vulcanization of test plates at 160 °C with time $t_{c90} + 2$ min
- Measurement of tensile strength and elongation at break with modulus 100 % 300 %,
In accordance with ISO 37: 2017-11
- Measurement of tear strength,
In accordance with DIN ISO 34-1: 2016-09, A
- Measurement of hardness Shore A,
In accordance with DIN ISO 7619-1: 2012-02
- Measurement of tension set after 5 hours at 105 °C and 50 % strain,
In accordance with DIN ISO 2285: 2010-06
- Determination of ageing resistance after 120 hours at 125 °C with measurement of:
 - tensile strength and elongation at break with modulus 100 % 300 %
 - tear strength
 - hardness Shore A
 - tension set after 5 hours at 105 °C and 50 % strain

4 Test results

Performance of the test:

Mixing	11.02.2020
Mooney viscosity	12.02.2020
Mooney scorch	13.02.2020
Heating	13.02.2020
Hardness, tensile strength, tear strength	18.02.2020
Ageing	20.02.2020 to 25.02.2020
Tension set	25.02.2020 to 28.02.2020

The following table (table 2) and the figures 1 and 2 are showing the compound properties in the uncured stage.

Table 2: Mooney and Mooney Scorch

Characteristic	MagPro 150	MgO Competitor 1	MgO Competitor 2
ML (1+4) 100 °C	46	45	45
Mooney scorch 125 °C			
Ts5 - minutes	28,8	27,9	27,8
Ts10 - minutes	41,2	37,0	38,3

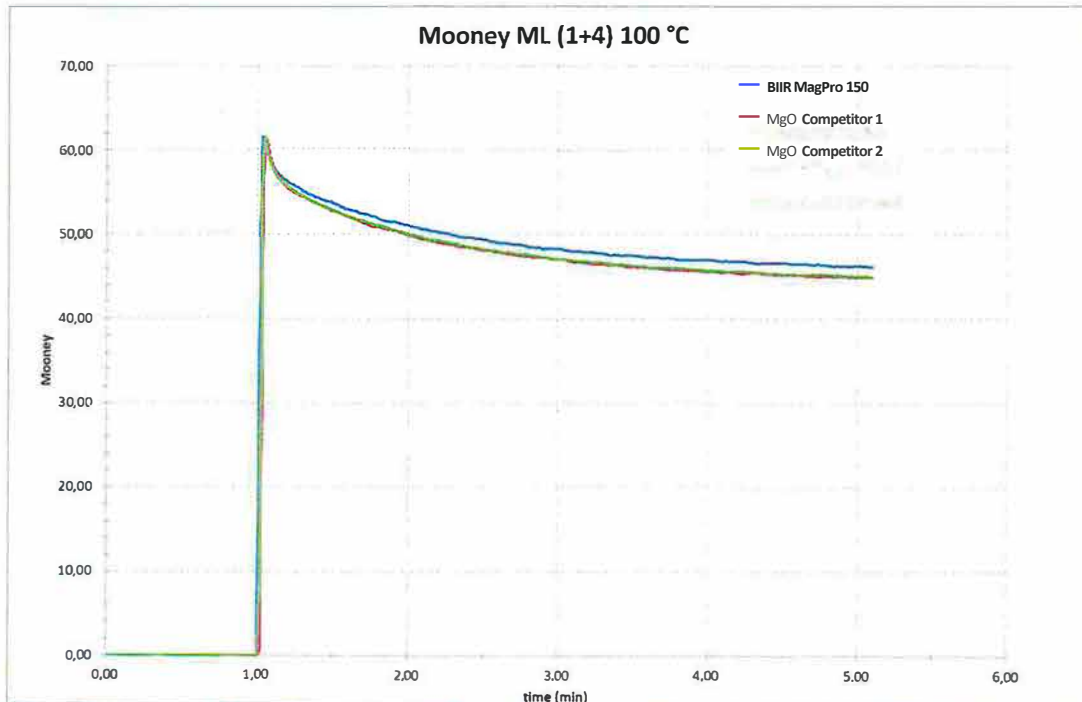


Figure 1.: Mooney viscosity at 100 °C

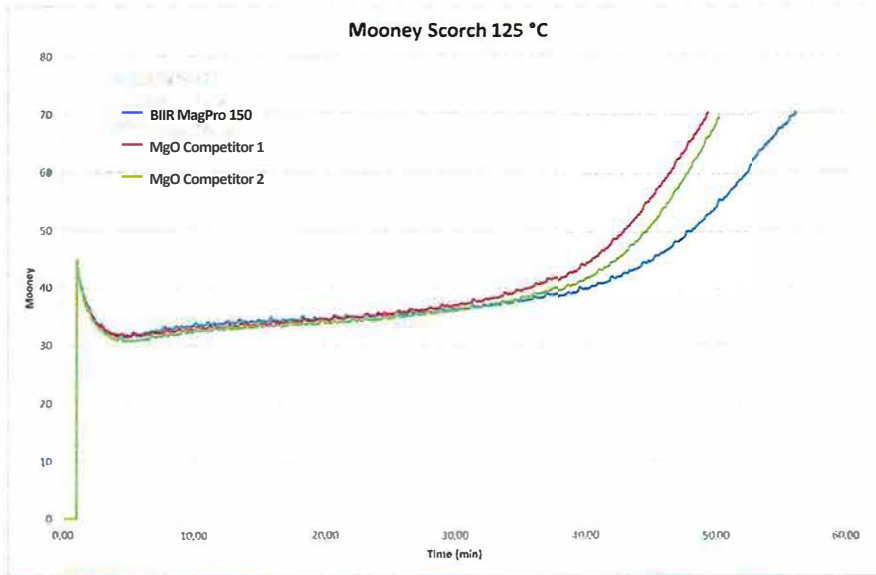


Figure 2.: Mooney Scorch at 125 °C

Table 3 and figure 4 are representing the cure characteristic of the investigated compounds.

Table 3: Rheometer 160°C, 60 minutes

Characteristic	MagPro 150	MgO Competitor 1	MgO Competitor 2
ML (Minimum torque) - dNm	1,21	1,18	1,16
MH (Maximum torque) - dNm	5,81	5,56	5,56
MH-ML (delta torque) - dNm	4,60	4,38	4,40
Ts2 - minutes	1,09	0,69	0,74
Tc25 - minutes	4,48	3,71	4,23
Tc90 - minutes	15,57	14,16	15,41

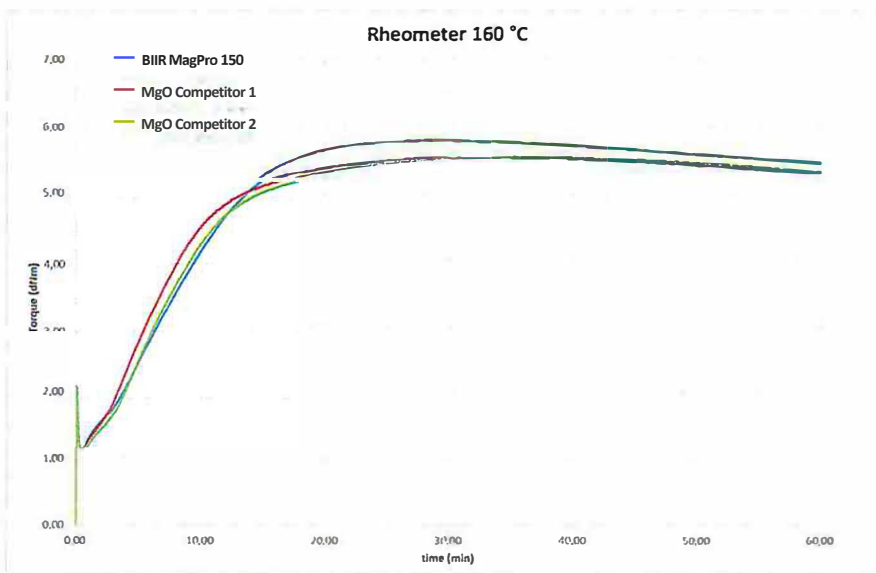


Figure 3: Rheometer at 160 °C

Table 4 gives the physical values for the vulcanized compounds.

Table 4.: Mechanical properties

Characteristic	MagPro 150	MgO Competitor 1	MgO Competitor 2
Tensile Strength - MPa	9,2 ± 0,7	9,3 ± 0,2	8,1 ± 0,4
Elongation at Break - %	572 ± 46	694 ± 15	584 ± 33
Modulus 100% - MPa	1,3 ± 0,1	1,3 ± 0,1	1,3 ± 0,1
Modulus 300% - MPa	4,8 ± 0,1	4,4 ± 0,1	4,5 ± 0,1
Tear Strength - KN/m	14,1 ± 0,4	15,1 ± 0,5	14,5 ± 0,5
Hardness – Units Shore A	49 ± 1	47 ± 1	48 ± 1
Tension Set - %	43,4 ± 0,6	47,1 ± 0,3	43,9 ± 0,4

The physical values of the investigated compound vary only concerning to the inaccuracy of the measurements. No significant difference is to be seen.

In table 5 the physical values after ageing for 120 hours at 125 °C are summarized for the vulcanized compounds.

Table 5: Mechanical properties after ageing 120 h at 125 °C

Characteristic	MagPro 150	MgO Competitor 1	MgO Competitor 2
Tensile Strength - MPa	7,7 ± 0,3	8,0 ± 0,1	6,7 ± 0,2
Elongation at Break - %	435 ± 17	526 ± 14	410 ± 22
Modulus 100% - MPa	1,8 ± 0,1	1,8 ± 0,1	1,8 ± 0,1
Modulus 300% - MPa	5,9 ± 0,2	5,7 ± 0,1	5,6 ± 0,1
Tear Strength - KN/m	9,6 ± 0,2	10,0 ± 0,2	9,6 ± 0,4
Hardness – Units Shore A	53 ± 1	51 ± 1	51 ± 1
Tension Set - %	30,2 ± 0,2	30,4 ± 0,3	29,8 ± 0,9

The aging-behaviors of the investigated compounds are mostly identical.

5 Summary

For the used BIIR compound all investigated MgO-types are comparable. The measured differences in cured and uncured state are not significant if one takes the measurements-inaccuracies into account. The scorch-characteristic of the MagPro 150 – compound seems to show a longer scorch times. The ageing behavior is more or less identical.

6 Experimentals

Table 6: Mixing recipe

		MagPro 150	MgO Competitor 1	MgO Competitor 2
Lfd. Nr.	Component	phr	phr	phr
1	Bromobutyl 2222	100,00	100,00	100,00
2	N 660	60,00	60,00	60,00
3	Vivatec 200	8,00	8,00	8,00
4	Tudalen 5138	7,00	7,00	7,00
5	Koresin	4,00	4,00	4,00
6	stearic acid	2,00	2,00	2,00
7	MagPro 150	0,50	---	---
8	MgO Competitor 1	---	0,5	---
9	MgO Competitor 2	---	---	0,5
10	ZnO RS	1,00	1,00	1,00
11	Sulfur	0,50	0,50	0,50
12	Vulkacit DM/ MBTS	1,50	1,50	1,50

Mixing of BIIR

Fill factor: 70 %

Start temperature: 40 °C

Rotor speed: 50 rpm

Mixing method: 0'00'' to 1'00'': Polymers

1'00'' to 2'00'': 2/3 Carbon black, ZnO, Stearic acid, MgO,
Tackifier, Antioxidants

2'00'' to 4'00'': 1/3 Carbon black, Oil

4'00'' to 5'00'': Sulfur, Accelerator

After dumping the compounds were homogenized on the rolling mill.

7 Comments

This test report has been created according to DIN EN ISO/IEC 17025. Results are shown as arithmetic means. More details can be given to the customer if requested.

Number of test samples: each n = 5

Before testing the samples have been stored for at least 24 hours under standard climate at 23 °C. While this range of time they were protected against the interfere of light.

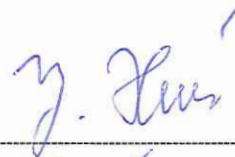
The preparation of the specimen, which are needed to perform standard test methods, was made by:

Punching from test plates with standard thickness.

8 Release

Person in charge: J. Heier

Head of testing: Dr. H. Geisler



Date: 02.03.2020

Stamp



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