# Comparison of Goodrich Flexometer tests:

# Test A: The inserted puncture needle is kept in the specimen throughout the entire test

# Test B: The puncture needle is inserted into the specimen immediately after the test

## 1 Introduction

The International Standard of Flexometer test ISO/CD 4666-4 prescribes the following method for the determination of the centre temperature of specimens: During the entire test the puncture needle is in the middle of the specimen and measures the centre temperature continuously.

In order to discover, whether the existence of the needle inside the specimen during the entire test has a disturbing effect on the centre temperature (e.g. by the friction heat during the deformation), this method is compared with the method of inserting the puncture needle into the specimen **immediately** after the test.

As a result the centre temperature is measured at the end of the test and no foreign substance is in the specimen during the test.

The tests were carried out with a fully automated DOLI Ultimate Flexometer, which fulfils the standards of ISO/CD 4666-4. The fully automated instrument guaranties an identical timing of all tests.

## 2 Material and Methods

The following described compounds and equipment are typically used in the rubber industry.

### 2.1 Compounds

Table 1 – Compounding of the samples

| **Compound characteristics:** | **NR** | **SBR** |
|---|---|---|
| **Ingredients:** | [phr] | [phr] |
| NR (RSS#1) | 100 | - |
| SBR 1502 | - | 100 |
| HAF Carbon black (N330) | 35 | 50 |
| ZnO | 5 | 3 |
| Stearic acid | 2 | 1 |
| 6PPD | 2 | 2 |
| TMDQ | 2 | 2 |
| Wax | 1 | 1 |
| TBBS | 0,7 | 1 |
| Sulphur | 2,25 | 1,75 |
| | | |
| **Total** | **149,75** | **161,75** |

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## 2.2 Instrument

For the comparison a fully automated DOLI Ultimate Flexometer was used. This instrument performs tests according to ASTM 623, ISO 4666-3 and ISO 4666-4 and guaranties exact timing of all the tasks. This means, the times for cool down and for pre-heat are kept accurately during all tests.

Functions of the puncture needle:

- Needle controlled in the centre of the specimen ( ISO/CD 4666-4)
- Insertion of the needle **immediately** after the test without loss of time. This function was the basis of the values in the discussion in October 2003 in London, too.

## 2.3 Puncture Needle

Puncture needle type: Type L (Fe-Cu-Ni); medium: silicon oil 150°C; insertion deepness: 12 mm; diameter of the needle: 1 mm. March of temperature see figure 1.

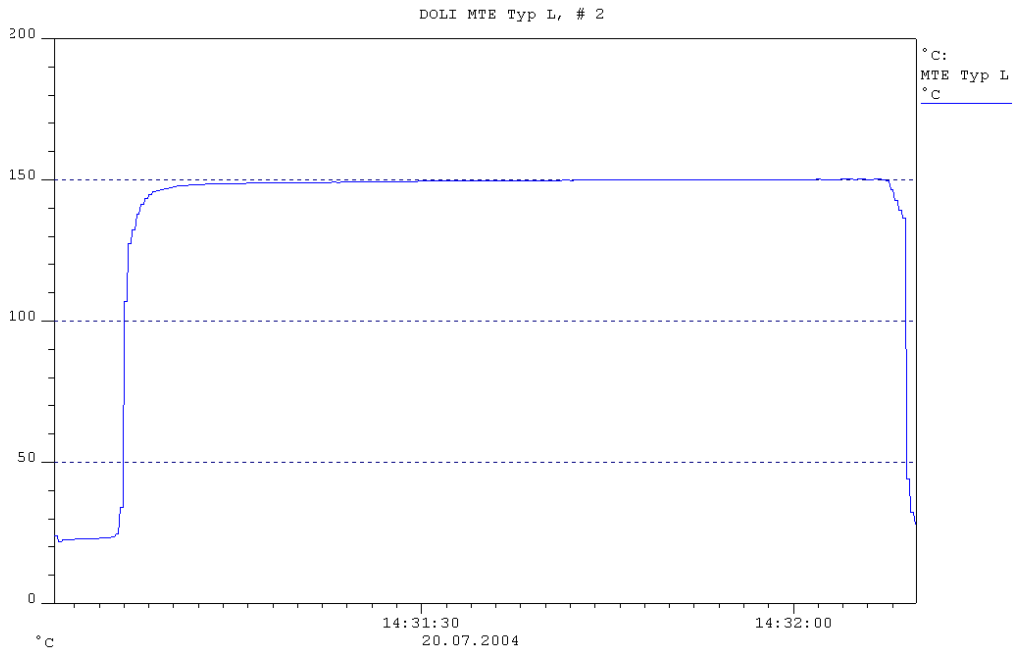


Fig.1: March of temperature for the used puncture needle.

## 3 Implementation of the Test

Test series A)

Two specimens of each of the compounds as stated (see 2.1.), are tested according to ISO/CD 4666-4. In order to measure the centre temperature, the needle is continuously controlled in the centre of the specimen and remains in it during the entire test.

Test series B)

For comparison, two specimens of each of the compounds as stated (see 2.1.), are tested with varied times of experimentation (1, 2, 5, 10, 15, 20 and 25 minutes). In order to measure the centre temperature, the puncture needle is inserted into the centre of the specimen **immediately** at the end of test.

Therefore, two new specimens are used for each time of experimentation. The temperature measurement takes place within the test area of the Flexometer. The time lag between dead stop of the instrument and the insertion of the needle is approx. 1 second. All the other test conditions of the standard test correspond to ISO/CD 4666-4.

Afterwards the specimen result charts (same components) are placed on top of each other and the values of centre temperature are compared (see 4. Fig. 2 and 3). For reasons of better overview, the curves of the deformation and the contact temperature of the specimen in test series B are not illustrated here. But the values are comparable. The result charts show the values of 16 specimens per chart.

# 4 Results

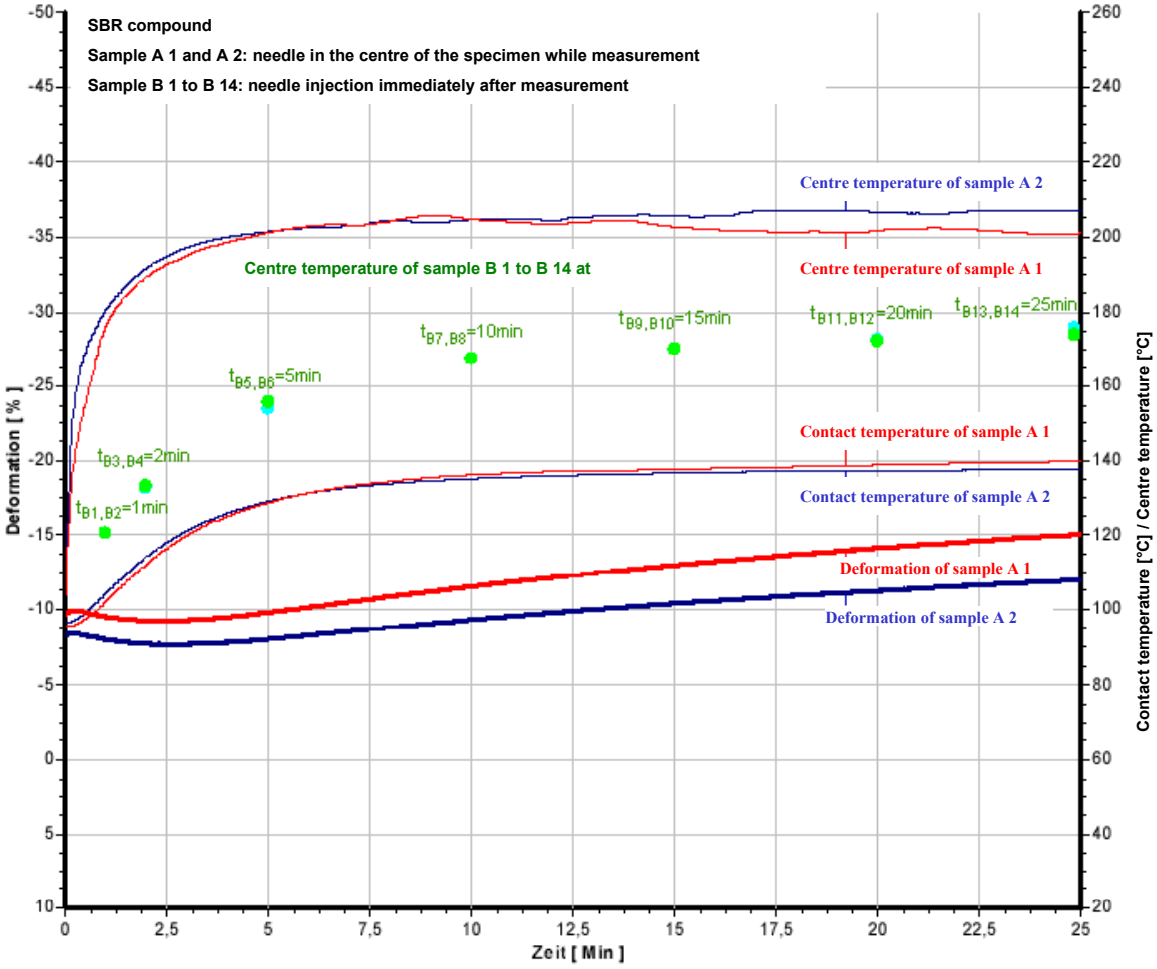


Fig.2: Comparison of the centre temperature for specimens of the compound SBR.

The samples A 1 (blue curves) and A 2 (red curves) are from the test series A. In this case the needle was in the centre of the specimen during the entire measurement. A continuous centre temperature curve was determined.

The samples B 1 to B 14 (green dots) are from the test series B. In this case the centre temperature was determined immediately after the measurement. For each test with a new test period two new test specimens were used; for these specimens, only the centre temperatures at the end of the tests are illustrated

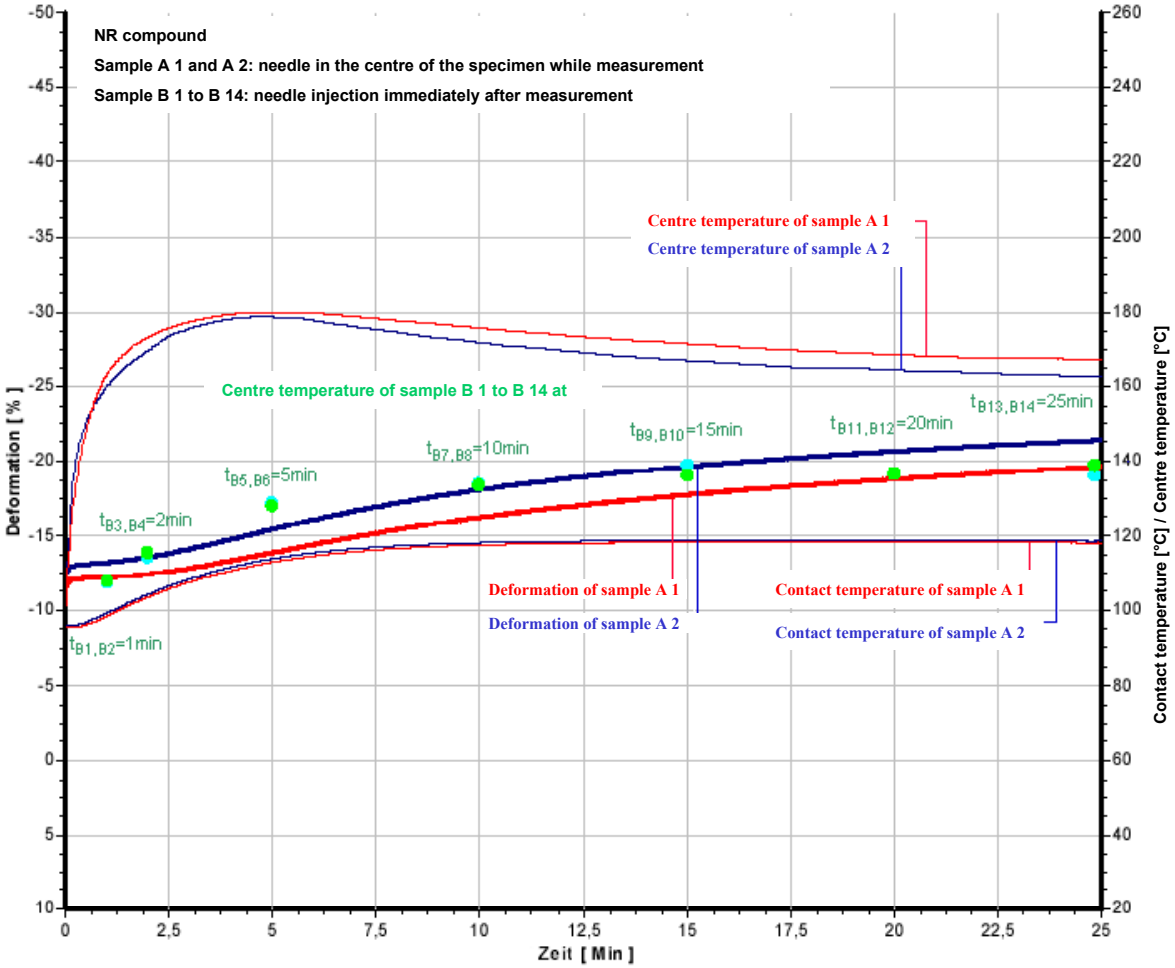


Fig.3: Comparison of the centre temperature for specimens of the compound NR.

The samples A 1 (blue curves) and A 2 (red curves) are from the test series A. In this case the needle was in the centre of the specimen during the entire measurement. A continuous centre temperature curve was determined.

The samples B 1 to B 14 (green dots) are from the test series B. In this case the centre temperature was determined immediately after the measurement. For each test with a new test period two new test specimens were used; the centre temperatures at the end of the tests are illustrated for these specimens, only.

1. Figures 2 and 3 show a clear difference in centre temperature between the two used methods. The measured centre temperatures for ‘test series A’ (needle remains continuously in the specimen) are distinctively higher than in ‘test series B’ (insertion of the needle after the end of the test).
2. The centre temperatures in ‘test series A’ increase distinctly at the beginning of the test. After that, they more or less remain constant and even drop. They do not run in parallel to the contact temperature.
3. The values of centre temperature in ‘test series B’ more or less run in parallel with the contact temperature.

Table 2: Centre temperatures of the test series A and B for the compounds SBR and NR with mean values and differences between the two series.

**SBR**

| Test time | Centre temperature [°C] | | | Centre temperature [°C] | | | |
|---|---|---|---|---|---|---|---|
| min | A1 | A2 | Mean value | Bx | By | Mean value | **Diff.** |
| 1,00 | 175,90 | 180,40 | 178,15 | 120,50 | 120,30 | 120,40 | **57,75** |
| 2,00 | 189,10 | 191,50 | 190,30 | 133,20 | 132,70 | 132,95 | **57,35** |
| 5,00 | 201,10 | 201,30 | 201,20 | 153,80 | 155,70 | 154,75 | **46,45** |
| 10,00 | 204,60 | 204,50 | 204,55 | 167,40 | 167,50 | 167,45 | **37,10** |
| 15,00 | 202,50 | 205,30 | 203,90 | 170,10 | 170,50 | 170,30 | **33,60** |
| 20,00 | 201,60 | 206,30 | 203,95 | 172,10 | 172,40 | 172,25 | **31,70** |
| 25,00 | 202,20 | 206,60 | 204,40 | 175,60 | 174,00 | 174,80 | **29,60** |

**NR**

| Test time | Centre temperature [°C] | | | Centre temperature [°C] | | | |
|---|---|---|---|---|---|---|---|
| min | A1 | A2 | Mean value | Bx | By | Mean value | **Diff.** |
| 1,00 | 162,90 | 159,80 | 161,35 | 107,60 | 108,10 | 107,85 | **53,50** |
| 2,00 | 173,10 | 169,80 | 171,45 | 116,50 | 115,50 | 116,00 | **55,45** |
| 5,00 | 179,90 | 178,40 | 179,15 | 128,70 | 127,80 | 128,25 | **50,90** |
| 10,00 | 178,80 | 176,30 | 177,55 | 133,50 | 134,10 | 133,80 | **43,75** |
| 15,00 | 171,40 | 167,10 | 169,25 | 138,60 | 136,10 | 137,35 | **31,90** |
| 20,00 | 168,60 | 164,20 | 166,40 | 136,40 | 136,40 | 136,40 | **30,00** |
| 25,00 | 167,90 | 162,50 | 165,20 | 136,20 | 138,50 | 137,35 | **27,85** |

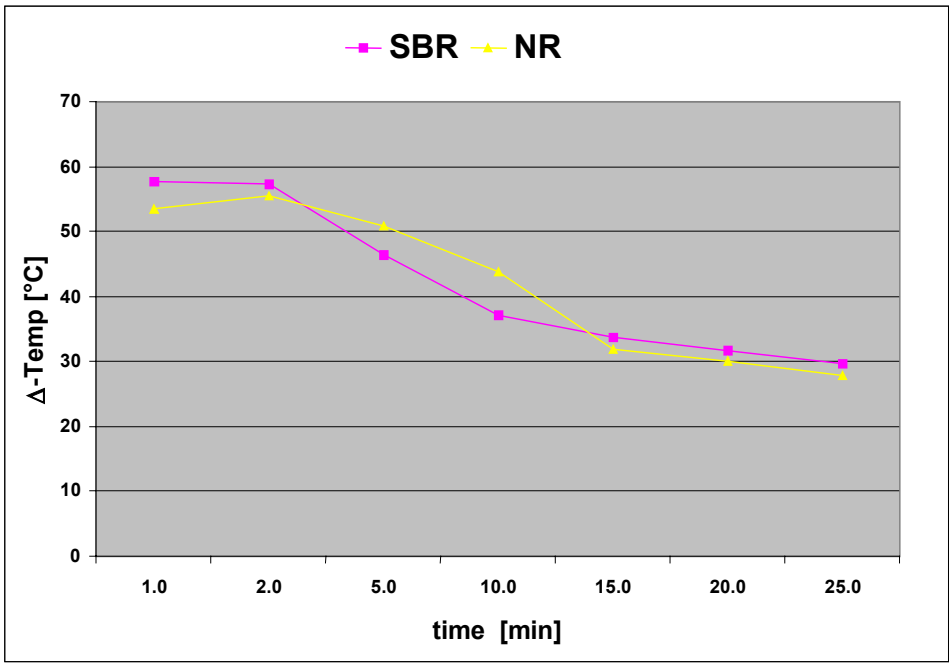


Fig.4: Differences in centre temperatures of the two test series A (test with needle inside the specimen) and B (test with temperature measurement immediately after test end) for the compounds SBR and NR.

The above chart (values from table 2) shows a big difference in centre temperature between the two test methods.

## 5 Discussion

1. There is a clear difference in the centre temperature between these two measurement methods. If the needle remains in the specimen during the test, the specimen will show a distinctly higher temperature than the specimen without a foreign substance in it. During the first 5 minutes a difference of approx. 50°C arises from this. After 15 minutes the difference levels out by 30°C, leading to the conclusion, that the existence of the needle falsifies the centre temperature in the specimen.

2. The behaviour of the course of the centre temperature in ‘test series A’ leads to the conclusion, that the increased temperature is the result of the friction between the specimen and the needle:

   The centre temperatures rise clearly within the first minutes of the test, but than remain relatively constant (fig. 2, SBR compound) or fall down distinctly (fig. 3, NR compound). The behaviour of the contact temperature is contrary, which leads to the conclusion, that at the start the friction on the needle is the greatest. Afterwards a lubrication or sliding at the needle occurs, which leads to a reduction of the measured centre temperature. This behaviour is dependent on the compound.

3. In contrast to this the measured values at the end of the test (test series B) run in parallel with the contact temperature every time, leading to the conclusion, that there is no disturbing factor.

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