



**Report**  
on Testing a Gasket Material for Reactivity with Oxygen

**Reference Number** 2-813/2013 II E

**Copy** 1. Copy of 2 Copies

**Customer** SGL CARBON GmbH  
Postfach 11 93  
86400 Meitingen  
Germany

**Order Date** March 1, 2013

**Receipt of Order** March 8, 2013

**Test Samples** SIGRAFLEX MF (V...11Z3MF), Batch  
12091014/11120868, for use as a gasket material in  
flanged connections in piping, valves and fittings or other  
components for gaseous oxygen service up to 130 bar  
and temperatures up to 250 °C;  
BAM Order-No. 2.1/51 485

**Receipt of Samples** March 8, 2013

**Test Date** May 24, 2013 to October 21, 2013

**Test Location** BAM - Working Group "Safe Handling of Oxygen";  
building no. 41, room no. 073 and no. 120

**Test Procedure or Requirement According to** DIN EN 1797: 2002-02  
„Cryogenic Vessels - Gas/Material Compatibility“  
ISO 21010: 2004-07  
„Cryogenic Vessels - Gas/Material Compatibility“  
Annex of pamphlet M 034-1 (BGI 617-1)  
"List of nonmetallic materials compatible with oxygen by BAM  
Federal Institute for Material Research and Testing.", by  
German Social Accident Insurance Institution for the raw  
materials and chemical industry,  
Edition: March 2013;  
Rule BGR 500 "Betreiben von Arbeitsmitteln" part 2,  
chapter 2.32 "Betreiben von Sauerstoffanlagen",  
paragraph 3.17 "Lubricants and sealing materials",  
Edition: April 2008.

All pressures of this report are excess pressures.  
This test report consists of page 1 to 5 and annex 1 to 3.

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In case a German version of the test report is available, exclusively the German version is binding.



## 1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test Application
- 1 Material Data Sheet  
(2 pages, revision no. 02 2009/0)
- 1 Safety Data Sheet  
(5 pages, revision no. 1.00, revised March 11, 2011)
- 15 Disks SIGRAFLEX MF (V... 11Z3MF), Batch 12091014/11120868  
Outer-Ø: 140 mm  
Color: Grey
- 3 Metal plates,  
coated with SIGRAFLEX MF (V... 11Z3MF), Batch 12091014/11120868  
Size: 145 mm x 145 mm; Thickness: 2 mm  
Color: Grey

## 2 Test Methods

To test and evaluate the compatibility of the sealing material SIGRAFLEX MF (V... 11Z3MF), Batch 12091014/11120868, for use as a gasket material in flanged connections in piping, valves and fittings or other components for gaseous oxygen service up to 130 bar and temperatures up to 250 °C, a flange test, the determination of the autogenous ignition temperature and an investigation of the aging resistance were carried out.

## 3 Test Results

### 3.1 Autogenous Ignition Temperature (AIT)

The test method is described in annex 1.

Results:

Test No.	Initial Oxygen Pressure $p_i$ [bar]	Final Oxygen Pressure $p_F$ [bar]	AIT [°C]
1	50	133	> 500
2	50	134	> 500
3	50	133	> 500
4	50	134	> 500
5	50	134	> 500

Up to temperatures of 500 °C, no ignition of the sealing material SIGRAFLEX MF (V... 11Z3MF), Batch 12091014/11120868, could be detected in five tests with initial oxygen pressures of  $p_i = 50$  bar. The final oxygen pressure  $p_F$  was approximately 133 bar.

### 3.2 Artificial Aging

The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	275	130	± 0

After aging of the sealing material SIGRAFLEX MF (V... 11Z3MF), Batch 12091014/11120868, at 130 bar oxygen pressure and 275 °C, the test sample was apparently unchanged. The sample mass did not change.

#### 3.2.1 AIT after Artificial Aging

The test method is described in annex 1.

Results:

Test No.	Initial Oxygen Pressure $p_i$ [bar]	Final Oxygen Pressure $p_f$ [bar]	AIT [°C]
1	50	134	> 500
2	50	131	> 500
3	50	133	> 500
4	50	133	> 500
5	50	133	> 500

Up to temperatures of 500 °C, no ignition of the sealing material SIGRAFLEX MF (V... 11Z3MF), Batch 12091014/11120868, could be detected in five tests with initial oxygen pressures of  $p_i = 50$  bar. The final oxygen pressure  $p_f$  was approximately 133 bar.

This shows, that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

### 3.3 Flange Test

The test method is described in annex 3.

Results:

Test No.	Oxygen Pressure [bar]	Temperature [°C]	Comments
1	130	250	Only those parts of the gasket burn that project into the pipe.
2	130	250	Same behavior as in test no. 1
3	130	250	Same behavior as in test no. 1
4	130	250	Same behavior as in test no. 1
5	130	250	Same behavior as in test no. 1

In five tests at 130 bar oxygen pressure and 250 °C, only those parts of the gasket burn that project into the pipe; the fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange remains gas-tight.

#### **4 Summary and Evaluation**

Up to temperatures of 500 °C, no ignition of the sealing material SIGRAFLEX MF (V...11Z3MF), Batch 12091014/11120868, could be detected with a final oxygen pressure  $p_F$  of approximately 133 bar.

At a temperature of 275 °C and an oxygen pressure of 130 bar, the sealing material SIGRAFLEX MF (V...11Z3MF), Batch 12091014/11120868, proved to be sufficient aging resistant. The sample mass did not change. Up to temperatures of 500 °C, no ignition of the aged sealing material SIGRAFLEX MF (V...11Z3MF), Batch 12091014/11120868, could be detected with a final oxygen pressure  $p_F$  of approximately 133 bar. This shows, that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

For safety reasons a safety margin of 100 °C between AIT and maximum operating temperature is being considered in evaluating nonmetallic materials for oxygen service. As the maximum operating temperature is 250 °C, the sealing material SIGRAFLEX MF (V...11Z3MF), Batch 12091014/11120868, fulfills this criterion.

On basis of those test results and the results of the flange testing, there are no objections with regard to technical safety to use the sealing material SIGRAFLEX MF (V...11Z3MF), Batch 12091014/11120868, as a gasket material in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at following conditions:

Maximum Temperature	Maximum Oxygen Pressure
250 °C	130 bar

This applies to flat faced flanges, male/female flanges, and flanges with tongue and groove.

This evaluation does not cover the use of the sealing material SIGRAFLEX MF (V...11Z3MF), Batch 12091014/11120868, for liquid oxygen service. For this case, a particular test for reactivity with liquid oxygen needs to be carried out.

#### **5 Comments**

The test results refer exclusively to the tested Batch 12091014/11120868 of the sealing material SIGRAFLEX MF (V...11Z3MF).

Products on the market that contain a reference to BAM testing shall be marked accordingly. It shall be evident that only a sample of a batch has been tested and evaluated for oxygen compatibility. The reference shall not produce a presumption of conformity that monitoring of the production on a regular basis is being performed by BAM.

It shall be clear that the product may only be used for gaseous oxygen service. The maximum safe oxygen pressure of the product and its maximum use temperature as well as other restrictions in use shall be given.

**BAM Federal Institute for Materials Research and Testing  
12200 Berlin, December 9, 2013**

**Division 2.1  
"Gases, Gas Plants"**

On behalf of



Dipl.-Ing. P. Hartwig  
Study Director "Safe Handling of Oxygen"

Copies:           1. Copy: SGL CARBON GmbH  
                      2. Copy: BAM – Division 2.1 "Gases, Gas Plants"

## Annex 1

### **Determination of the Autogenous Ignition Temperature in High Pressure Oxygen**

A mass of approximately 0.1 g to 0.5 g of the pasty or of the divided solid sample is placed into an autoclave (34 cm<sup>3</sup> in volume) with a chrome/nickel lining. Liquid samples are applied onto ceramic fiber.

The autoclave is pressurized to the desired pressure  $p_a$  at the beginning of the test. A low-frequency heater inductively heats the autoclave in an almost linear way at a rate of 110 K/min. The temperature is monitored by means of a thermocouple at the position of the sample.

The pressure in the autoclave is measured by means of a pressure transducer. Pressure and temperature are recorded. During the test, as the temperature increases, the oxygen pressure increases within the autoclave. The ignition of the sample can be recognized by a sudden rise in temperature and pressure. The oxygen pressure on ignition  $p_o$  is calculated.

It is important to know the oxygen pressure  $p_o$ , as the autogenous ignition temperature of a material is a function of pressure. It may decrease as the oxygen pressure increases.

## **Annex 2**

### **Testing for Aging Resistance in High Pressure Oxygen**

A sample with known mass is exposed to high-pressure oxygen at elevated temperature in an autoclave for 100 hours. The temperature, at which the sample is aged, is at least 100 °C lower than the autogenous ignition temperature of the sample.

This test shows whether the sample gradually reacts with oxygen or whether it undergoes other visible changes. If there is no change in appearance, in mass, and in the autogenous ignition temperature of the material, it is considered aging resistant.

### **Annex 3**

#### **Testing of Gaskets for Flanges in Oxygen Steel Pipings**

The test apparatus mainly consists of two DN 65 PN 160 steel pipes, each approximately 2 m in length, with corresponding standard flanges welded to each pipe.

Both pipes are sealed using the gasket to be tested. In case of a gasket disk its inner diameter is chosen in such a way that it projects into the pipe. If a gasket tape is under test, both ends of the tape are allowed to project into the pipe. The test apparatus is then pressurized with oxygen up to the desired test pressure. The flange is heated by heating sleeves to the test temperature, at least 50 K lower than the ignition temperature of the gasket. An electrical filament ignites that part of the gasket projecting into the pipe. If the gasket is electrically conductive, such as spiral seals or graphite foils, a nonconductive primer capsule of organic material (PTFE, rubber) is used which acts on the seal.

The gasket's behavior after ignition is important for its evaluation. If the seal burns with such a hot flame that the fire is transmitted to the steel of the flange (in most case the test apparatus is destroyed), the seal is considered unsuitable from the beginning. If only those parts of the seal burn that project into the pipe and the fire is not transmitted to the flanges and if the seal does not burn between the flanges there are no objections with regard to technical safety to use the seal under the conditions tested. Such a positive result is to confirm in four additional tests. If, however, the flanged connection becomes un-tight during a test, e. g., because of softening or burning of the seal, the test has to be continued at a lower temperature and oxygen pressure until a positive test result is reached in five tests, as mentioned above.