BAM
Bundesanstalt für
Materialforschung
und -prüfung

TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

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Reference Number	17063170 E
Our Reference	02-3473
Copy	1. copy of 2 copies
Customer	Frenzelit GmbH Frankenhammer 7 95460 Bad Berneck Germany
Date of Request	November 23, 2017
Your Reference	EMP / BWI
Receipt of Signed Contract	January 3, 2018
Test Samples	Gasket material novapress® 880, batch 29114569133;
Receipt of Samples	December 6, 2017
Test Date	January 9 to 25, 2018
Test Location	BAM – Division 2.1 „Gases, Gas Plants"; building no. 41
Test Procedure or Requirement according to (in the current version)	DIN EN 1797 und ISO 21010 "Cryogenic Vessels - Gas/Material Compatibility"; Annex of code of practice M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen"

All pressures of this report are excess pressures.
This test report consists of page 1 to 9 and annex 1 to 3.

TEST REPORT

1 Documents and Test Samples

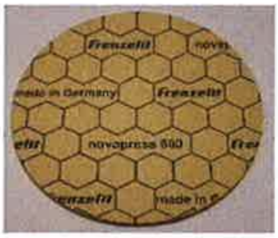
The following documents and samples were submitted to BAM:

1 Test application
Safety-related investigation on the material novapress® 880, batch 29114569133, for use as a gasket material in flanges for gaseous oxygen at temperatures up to 80 °C and at pressures up to 120 bars

1 Safety Data Sheet novapress® 280, 805, FLEXIBLE/815, 820, 850, 880, AKTIV, BASIC
(6 pages, Fa. Frenzelit Werke GmbH, Version 1.5, date of issue: 02.05.2017)

1 Material Data Sheet novapress® 880
(1 page, Version 1, No. 01608, date of issue: 13.09.2017)

15 Disks novapress® 880, batch 29114569133
Single-sided imprint "Frenzelit, novapress® 880, Made in Germany"
Dimensions: Outer diameter: 140 mm, Thickness: 3 mm
Color: Beige



2 Applied Test Methods

The product novapress® 880, batch 29114569133, shall be used as a gasket material in flanges for gaseous oxygen service at temperatures up to 80 °C.

The following test methods were applied:

2.1 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

Usually, this test method is required if the material is for service temperatures greater than 60 °C.

The AIT is a safety characteristic and indicates the temperature at which the material shows self-ignition in the presence of oxygen without an additional ignition source. For gasket materials in flange connections, the safety margin between AIT and maximum use temperature is only 50 °C because of the particular mounting situation.

2.2 Testing for Aging Resistance in High Pressure Oxygen

This test is necessary whenever a material is intended for service at higher temperatures than 60 °C. It simulates the use of a material in practice and helps analyze whether ignition temperature or properties of the material change due to the aging processes.

2.3 Testing of Gaskets for Flanges in High Pressure Oxygen

This test simulates the faulty installation of a gasket in a flange connection where the sealing material projects into the inner diameter of the pipe. This test investigates the fire behavior of the gasket material in a standard flange after artificial ignition. It shows whether the fire of the disk is transferred to the metal of the flange or if the flange connection becomes leaky.

3 Preparation of Samples

To test the nonconductive gasket material, the disks were prepared as shown in figure 1.

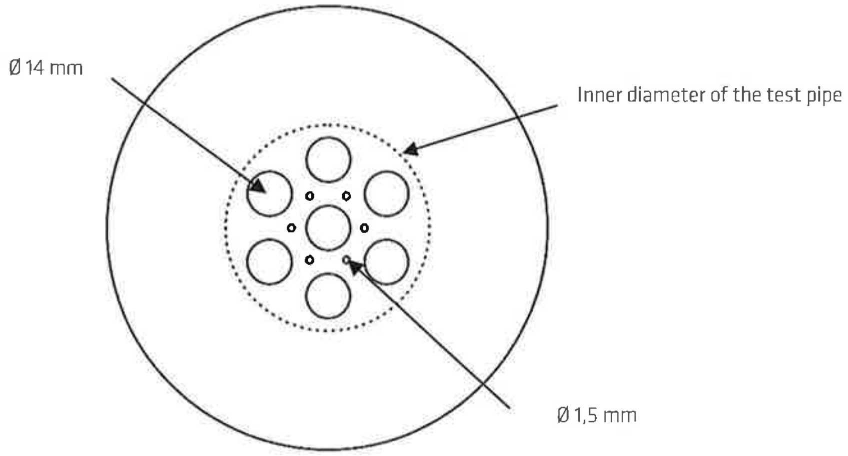


Figure 1: Preparation of the nonconductive flat gasket material

For all other tests, the disks were cut into parts of ca. 1 mm to 2 mm in edge length.

4 Tests

4.1 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

The test method is described in annex 1.

The AIT determination was performed at a final oxygen pressure of approximately 120 bars according to the intended use conditions mentioned by the customer.

4.1.1 Assessment Criterion

The criterion for a reaction of the sample with oxygen is a distinct increase in pressure and a more or less steep increase in temperature.

4.1.2 Results

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	87	125	147
2	87	127	150
3	87	127	151
4	87	127	147
5	87	126	144

In five separate tests, the following mean AIT could be determined:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
126	148	± 3

4.2 Testing for Aging Resistance in High Pressure Oxygen

The test method is described in annex 2.

In general, artificial aging is carried out at the maximum use pressure and an elevated temperature, that is 25 °C above the maximum operating temperature. In this case, the test was carried out at a final oxygen pressure of 120 bars and at a temperature of 105 °C.

4.2.1 Assessment Criterions

There are three criteria for evaluating the aging behavior:

If there is a change in mass $\Delta m \leq 1$ %, the sample is aging resistant, in case of $\Delta m > 1$ % and $\Delta m \leq 2$ %, the sample is sufficient aging resistant, and in case of $\Delta m > 2$ %, the sample is insufficient aging resistant.

Changes in color, consistency, shape or surface texture of the sample or gas releases from the sample that can be detected after testing will be also considered by BAM.

The AIT of the aged sample is compared to the AIT of the non-aged sample. If there is a distinct deviation between both AITs, the lower value is considered for safety reasons.

4.2.2 Results

4.2.2.1 Testing for Change in Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	105	120	+ 1.7

After aging, an intensive malodor was noted and the test sample was highly brittle. In addition, the sample increased 1.7% in mass.

4.2.2.2 Determination of the AIT of the Aged Material in High Pressure Oxygen

The test method is described in annex 1.

The AIT test of the aged material was performed under the same conditions as described in chapter 4.1.

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	87	138	190
2	87	139	188
3	87	138	187
4	87	138	191
5	87	136	178

In five separate tests, the following mean AIT could be determined:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
138	187	± 5

4.3 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 3.

4.3.1 Assessment Criterion

If after artificial ignition only those parts of the gasket burn that project into the pipe and the fire is not transmitted to the flanges, and if the gasket does not burn between the flange faces and the flange connection is still gas tight, there are no objections regarding technical safety to use the gasket under the conditions tested. Such a positive result has to be confirmed in four additional tests.

If, however, the gasket burns between the flange faces or the flange connection becomes un-tight, the gasket material has not passed the test. In this case, the test may be continued at a lower temperature or oxygen pressure after consultation with the customer.

4.3.2 Results

Test No.	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	80	120	All parts of the gasket burn that project into the pipe and the material also burns between the flange surfaces. The flange faces remain undamaged. The flange connection remains gas-tight.

Already in the first test at a temperature of 80 °C and at a final oxygen pressure of 120 bars, not only those parts of the gasket burn that project into the pipe but the material also burns between the flange surfaces.

After consulting the customer, the tests were continued at a reduced oxygen pressure of 110 bars.

Test No.	Temperature [°C]	Oxygen Pressure [bar]	Notes
2	80	110	All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas-tight
3	80	110	Same behavior of test sample as in test no. 2
4	80	110	Same behavior of test sample as in test no. 2
5	80	110	Same behavior of test sample as in test no. 2
6	80	110	Same behavior of test sample as in test no. 2

In five tests at 80 °C and at a final oxygen pressure of 110 bars, only those parts of the disk burn that project into the pipe. In all tests, the fire is neither transmitted to the steel nor does the sample burn between the flange faces. The flange remains gas-tight. After testing, the samples exhibit a thickness of approximately 2.9 mm.

5 Summary of the Test Results

At a final oxygen pressure p_F of 126 bars, the sample has an autogenous ignition temperature of 148 °C with a standard deviation of ± 3 °C.

The material is not aging resistant at 105 °C and 120 bars oxygen pressure, as the material became very brittle after aging.

The investigation of the burning behavior of disks of the gasket material in a standard flange showed that at 80 °C and an oxygen pressure of 110 bars only those parts of the sample burn that project into the pipe. The sample does not burn between the flange faces. In all cases the flange connection remained gas-tight.

6 Opinion and Interpretation

The product novapress® 880, batch 29114569133, shall be used as a gasket material in flanges on components for gaseous oxygen.

On basis of the test results, the requirements for sealing materials, described in annex 1 to attachment 2 of code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and BAM´s safety philosophy, there are no objections regarding technical safety, to use the material novapress® 880, batch 29114569133, as a gasket material with a maximum thickness of 3 mm in flange connections made of copper, copper alloys or steel for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
80	110

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

However, we would like to point out, that the unfavorable aging behavior of the material may have an influence on its practical usability.

7 Comments

This safety evaluation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure impacts - can be safely excluded on the material in usage.

This evaluation is based exclusively on the results of the tested sample of a particular batch.

Our experience shows, that the safety characteristics of a product may vary from batch to batch. Therefore, today, we recommend batch testing of products, that are included for oxygen service. In this context, we would like to mention our paper from September 2009: "The Importance of Quality Assurance and Batch Testing on Nonmetallic Materials Used for Oxygen Service", Journal of ASTM International, Vol. 8th; Paper ID JAI102309. This publication can be purchased at www.astm.org.

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.

The product may 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.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

February 6, 2018

Division 2.1 "Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list: 1. copy: Frenzelit GmbH
2. copy: BAM - Division 2.1 "Gases, Gas Plants"

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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^3 in volume) with a chrome/nickel lining. Liquid samples are applied onto ceramic fiber.

The autoclave is pressurized to the desired initial pressure p_I 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 the final pressure p_F.

It is important to know the oxygen pressure p_F, 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.

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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.