BAM
Bundesanstalt für Materialforschung und -prüfung

TEST REPORT

BAM reference	22049596-I-E
Copy	1st copy of 2
Customer	Rich. Klinger Dichtungstechnik GmbH & Co. KG Am Kanal 8-10 2352 Gumpoldskirchen Austria
Order Date	December 5, 2022 and March 6, 2023
Your Reference	-
Receipt of Contract	January 31, 2023
Test Samples	Centellen® WS 3820, batch 72616
Receipt of Samples	December 1, 2022
Test Date	December 1, 2022, to March 29, 2023
Test Location	Division 2.1 "Safety of Energy Carriers"; building no. 41
Test Procedure or Requirement according to	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"

This test report consists of page 1 to 8 and enclosures 1 to 3

1 Documents and Test Samples

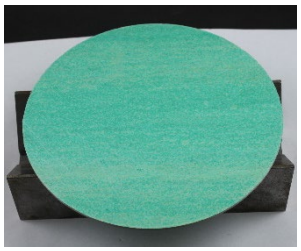
The following documents and samples were submitted to BAM:

1 Test application
Safety-related investigation on gasket material Centellen® WS 3820, batch 72616, for use in gaseous oxygen service at temperatures up to 80 °C and pressures up to 100 bars

1 Sicherheitsinformation Centellen® WS 3820
(6 pages, Rich. Klinger Dichtungstechnik GmbH & Co. KG, date of issue: 07.11.2022)

1 Completely filled Customer Master Data Sheet (CMDS) (November 23, 2022)

16 Disks of gasket material Centellen® WS 3820, batch 72616
Dimensions: Ø 140 mm, Thickness 2 mm
Color: Turquoise



2 Applied Test Methods

The gasket material Centellen® WS 3820, batch 72616, shall be used for gaseous oxygen service at temperatures up to 80 °C and at pressures up to 100 bars. Based on the information by the customer, oxygen pressure impacts on the material can be safely excluded in usage.

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. Therefore, it is relevant for the maximum use temperature, that is generally set 50 °C below this AIT for gasket materials.

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 Sampling

The material sample used for the investigation was provided by the customer.

3.1 Preparation of Samples

For testing the "Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen", the gasket material Centellen® WS 3820, batch 72616, was cut into parts of ca. 1 mm to 2 mm in edge length and was used in this form.

For testing the "Aging Resistance in High Pressure Oxygen", the gasket material Centellen® WS 3820, batch 72616, was cut into parts of ca. 40 mm to 20 mm in edge length and was used in this form.

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

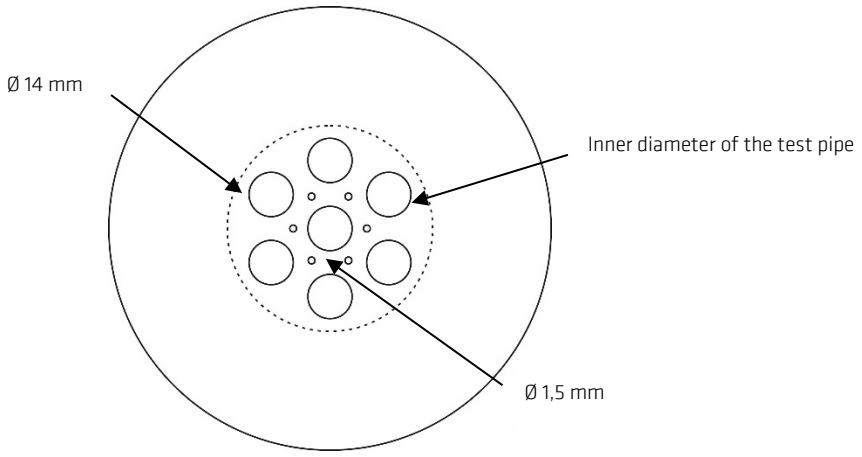


Figure 1: Preparation of the nonconductive flat gasket material

4 Tests

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

The test method is described in annex 1.

Based on the intended use pressure, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 100 bars.

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.	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	101	158
2	103	158
3	102	155
4	103	158
5	103	157

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

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
102	157	± 1

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 at an elevated temperature, that is 25 °C above the maximum operating temperature. Consequently, the test temperature was 105 °C. Based on the intended use pressure, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 100 bars.

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	100	+ 4.9

After aging, the test sample was embrittled, slightly discolored and had a strong smell. The test sample increased 4.9 % in mass.

The test sample did not pass the “Test for Aging Resistance in High Pressure Oxygen”.

After consultation the customer, and in deviation to the original test request and order, BAM was informed by email on March 6, 2023, that the material should only be tested for an operating temperature of 60 °C. In this case only the "Test of Gaskets for Flanges in High Pressure Oxygen" is carried out at 60 °C.

Consequently, the determination of the Autogenous Ignition Temperature (AIT) of the aged material was not carried out.

4.3 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 3.

Based on the intended use pressure and on the lowered use temperature, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 100 bars and at a temperature of 60 °C.

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 Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	60	100	All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas tight.
2	60	100	Same behavior as in test no. 1
3	60	100	Same behavior as in test no. 1
4	60	100	Same behavior as in test no. 1
5	60	100	Same behavior as in test no. 1

In five tests at 60 °C and an oxygen pressure of 100 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 the tests, the tested samples had a thickness of 2 mm.

5 Summary of the Test Results

At a mean final oxygen pressure p_F of 102 bars, the test sample has a mean autogenous ignition temperature of 157 °C with a standard deviation of ± 1 °C.

The material is insufficient aging resistant at 105 °C and at 100 bars oxygen pressure.

The investigation of the burning behavior of disks of the gasket material in a standard flange showed that at 60 °C and at an oxygen pressure of 100 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 Measurement uncertainty

The tests are carried out in accordance with the standards or guidelines indicated on the cover sheet of this report. Thereafter, the temperature measurement should have a maximum deviation of ± 2 K and the pressure measurement should have a maximum deviation of ± 2 bar.

For the test in chapter 4.1, the extended uncertainty is 0.7 K (according to the calibration protocol from January 23, 2023) for the temperature measuring system, and the uncertainty is 0.3 bar (according to the calibration protocol from January 25, 2023) for the used pressure measuring system.

For the test in chapter 4.2, the extended uncertainty is 1.0 K (according to the calibration protocol from February 2023) for the temperature measuring system, and the uncertainty is 0.3 bars (according to the calibration protocol from February 2023) for the used pressure measuring system.

For the test in chapter 4.3, the extended uncertainty is 1.0 K (according to the calibration protocol from February 27, 2023) for the temperature measuring system, and the uncertainty is 0.7 bars (according to the calibration protocol from September 8, 2022) for the used pressure measuring system.

7 Statements of conformity

The tests are carried out in accordance with the standards or guidelines, stated on the cover sheet of this report. Deviating or supplementary test criteria are described in the respective subchapter "Assessment Criterion" in Chapter 4 "Tests".

8 Opinion and Interpretation

The gasket material Centellen® WS 3820, batch 72616, shall be used in gaseous oxygen service at temperatures up to 60 °C and at pressures up to 100 bars. Based on the information by the customer, oxygen pressure impacts on the material can be safely excluded in usage.

On basis of the test results, the requirements for sealing materials, described in the code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and based on the assessment criteria described in this test report, there are no objections regarding technical safety, to use the gasket material Centellen® WS 3820, batch 72616, for gaseous oxygen service at following use conditions only:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	100

9 Comments

This safety-related investigation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges – can be safely excluded in usage. In addition, the safety related investigation considers the fact, that the material shall be used in gaseous oxygen service at temperatures up to 60 °C.

The content of the test report refers exclusively to the test sample of the gasket material Centellen® WS 3820, batch 72616.

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 the specified purpose in gaseous oxygen service only. 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

May 8, 2023

Division 2.1 "Safety of Energy Carriers"

by order

Dr. Thomas Kasch	Dr. Kai Holtappels
Study Director	Head of Division

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Enclosure 1

Determination of the Autogenous Ignition Temperature of Nonmetallic Materials in High Pressure Oxygen (V 2023-01)

0.2 g to 0.5 g of the paste-like, of the divided solid, or with ceramic fibres mixed liquid material is placed into an autoclave with a volume of 34 cm^3. The autoclave is pressurized to the initial pressure p_i and inductive heated. The temperature increases in an almost linear way at a rate of 110 K/min.

Pressure and temperature are recorded by a PC-system. As the temperature increases, the oxygen pressure in the autoclave increases continuously. The ignition of the material is recognized by a sudden rise in temperature and a more or less rise of the pressure.

In this way, the ignition temperature is determined at a specific final oxygen pressure p_f. In principle, the ignition temperature of a material depends on the pressure. The ignition temperature decreases with increasing final oxygen pressure.

For this test, the maximum test pressure is 250 bars, and the maximum test temperature is 500 °C.

Enclosure 2

Testing for Aging Resistance of nonmetallic materials in High Pressure Oxygen (V 2023-01)

A sample of the solid material is exposed to compressed oxygen and elevated temperature in an autoclave for 100 hours. The sample mass is determined before the test.

This test is intended to simulate the use of the material in practice and to show whether the material properties such as color, consistency, surface texture or the ignition temperature of the material change as a result due to aging processes.

For this test, the maximum test pressure is 250 bars, and the maximum test temperature is 325 °C.

Enclosure 3

Testing Nonmetallic Gaskets for Flanges in Oxygen Steel Pipings (V 2023-01)

The test facility mainly consists of two DN 65 PN 160 steel pipes, each 2 m in length, with corresponding standard flanges welded to each pipe. The customer provides the nonmetallic gasket material in form of disks. Using this disk, the standard flanges are flanged gas tight.

The test facility is heated to the intended maximum use temperature and pressurized to the intended maximum use pressure with oxygen. Thereafter, the part of the nonmetallic gasket material is ignited, which projects into the pipe.

The nonmetallic gasket material passes the test, if only that part of the gasket burns those projects in to the pipe, the material does not burn between the flange faces more than 2 mm, and the flange connection is still gas tight. The test is finished, if there is no reaction of the material detected in five single tests.

If a reaction occurs and after consultation the customer, testing the nonmetallic gasket material can be continued for use at lower operating conditions.

For this test, the maximum test pressure is 160 bars, and the maximum test temperature is 300 °C.