BAM
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und -prüfung

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TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

Reference Number	17027051 E
Our Reference	02-3419
Copy	1. copy of 2 copies
Customer	Garlock GmbH Falkenweg 1 41468 Neuss Germany
Date of Request	May 8, 2017
Your Reference	GYL3500 KU 2017
Receipt of Signed Contract	June 20, 2017
Test Samples	Gasket material GYLON® Style 3500, undisclosed batch;
Receipt of Samples	July 5 and September 1, 2017
Test Date	August 2 to September 25, 2017
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 10 and annex 1 to 4.

TEST REPORT

1 Documents and Test Samples

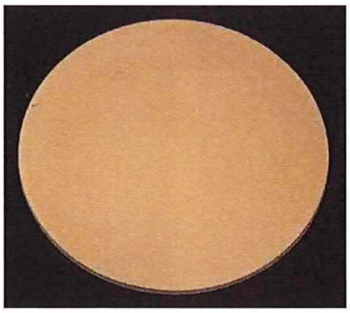
The following documents and samples were submitted to BAM:

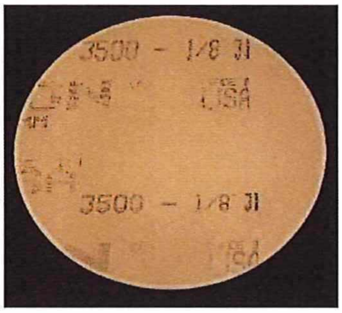
1 Test application
"Safety-related investigation of the flat gasket material GYLON® Style 3500, undisclosed batch, for flanges in gaseous oxygen service at pressures up to 83 bar and at temperatures up to 260 °C and for liquid oxygen service"

1 Safety Data Sheet GYLON® Style 3500
(7 pages, Garlock Sealing Technologies, date of issue: July 31, 2006)

1 Material Data Sheet Garlock FAWN GYLON® Style 3500
(1 page, Garlock Sealing Technologies)

30 Disks GYLON® Style 3500, undisclosed batch,
Single-sided imprint:"3500 – 1/8 31, Made in USA"
Dimensions:
Diameter 140 mm, Thickness 3.2 mm
Color: Fawn





2 Applied Test Methods

The product GYLON® Style 3500, undisclosed batch, is a flat gasket material intended for use in flanges on components for gaseous oxygen service at temperatures up to 260 °C and for liquid oxygen service.

Reactivity testing of GYLON® Style 3500, undisclosed batch, to rapid oxygen pressure changes, so-called oxygen pressure impacts, was not carried. According to the information by the customer, oxygen pressure impacts can be safely excluded in the intended service.

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 sealing 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 of the Aging Behavior 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.

2.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

Generally, this test method is required if direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage.

3 Preparation of Samples

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

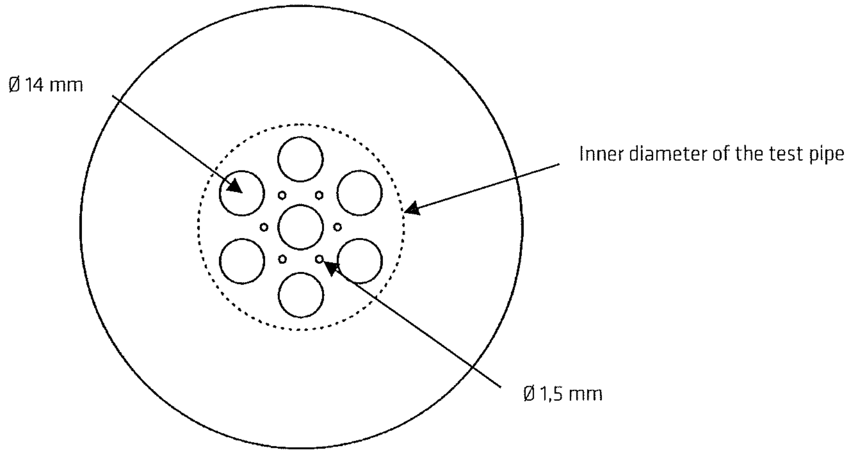


Figure 1: Preparation of the nonconductive flat gasket material

For all other tests, the disks were cut into parts of ca. 1 mm^3 to 2 mm^3 in size.

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 83 bar 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	33	83	461
2	33	83	457
3	33	83	457
4	33	83	458
5	33	83	456

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

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
83	458	± 2

4.2 Testing of the Aging Behavior 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 83 bar and a temperature of 285 °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 Change in Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	285	83	- 0,1

After aging, the test sample was apparently unchanged and lost 0,1 % 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	33	83	463
2	33	83	455
3	33	83	456
4	33	83	459
5	33	84	467

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

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
83	460	± 5

4.3 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 3.

In contrast to the request of the customer, and on basis of BAM's experience with sealing materials that consist of similar constituents as the test sample, flange testing started first at a final oxygen pressure of 40 bar instead of 83 bar at a temperature of 260 °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	260	40	All parts of the gasket burn that project into the pipe. The sample burns up to 4 mm between the flange faces. The flange connection remains gas-tight.
2	260	30	All parts of the gasket burn that project into the pipe. The sample burns up to 3.2 mm between the flange faces. The flange connection remains gas-tight.
3	260	25	All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas-tight.
4	260	25	Same behavior as in test no. 3
5	260	25	Same behavior as in test no. 3
6	200	40	All parts of the gasket burn that project into the pipe. The sample burns up to 2.6 mm between the flange faces. The flange connection remains gas-tight.
7	200	25	Same behavior as in test no. 3
8	200	25	Same behavior as in test no. 3
9	100	40	All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas-tight.

Test Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
10	100	40	Same behavior as in test no. 9
11	100	40	Same behavior as in test no. 9
12	100	40	Same behavior as in test no. 9
13	100	40	Same behavior as in test no. 9

In three tests at 260 °C and accordingly in two tests at 200 °C and an oxygen pressure of 25 bar at each temperature, only those parts of the disk burn that project into the pipe.

In five tests at 100 °C and an oxygen pressure of 40 bar, 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. The tested samples keep almost their original thickness.

4.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

4.4.1 Assessment Criterion

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", a nonmetallic material is not suitable for liquid oxygen service, if reactions occur with liquid oxygen at a drop height of 0.17 m (impact energy 125 Nm) or less.

4.4.2 Results

Test No.	Drop Height [m]	Impact Energy [Nm]	Behavior on Mechanical Impact
1	0,67	500	reaction
2	0,50	375	reaction
3	0,33	250	reaction
4	0,17	125	reaction
5	< 0,17	< 125	reaction

At a drop height of 0,67 m, 0,50 m, 0,33 m, 0,17 m and below (impact energies 500 Nm, 375 Nm, 250 Nm 125 Nm, and < 125 Nm) reactions of the sample with liquid oxygen could be regularly detected - to some extent under the formation of sparks.

5 Summary of the Test Results

At a final oxygen pressure p_F of 83 bar, the test sample has an autogenous ignition temperature of 458 °C with a standard deviation of ± 2 °C.

The material proved to be aging resistant at 285 °C and 83 bar oxygen pressure.

The investigation of the burning behavior of disks of the gasket material in a standard flange showed that at 260 °C and 200 °C and an oxygen pressure of 25 bar only those parts of the sample burn that project into the pipe. The sample does not burn between the flange faces. The same burning behavior of the disks could be observed at 100 °C and 40 bar oxygen pressure. In all cases the flange connection remained gas-tight.

Testing of the material for reactivity to mechanical impacts in liquid oxygen showed that always reactions occurred, even at an impact energy of less than 125 Nm.

6 Opinion and Interpretation

The product GYLON® Style 3500, undisclosed batch, shall be used as a flat 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 nonmetallic flat gasket material GYLON® Style 3500, undisclosed batch, with a maximum thickness of 3,2 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]
100	40
> 100 up to 200	25

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

Based on the test results and according to BAM' s standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", the gasket material GYLON® Style 3500, undisclosed batch, <u>is not suitable</u> for liquid oxygen service.

7 Comments

This safety-related investigation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges – can be safely excluded 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, 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

November 30, 2017

Division 2.1 "Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list:	1. copy:	Garlock 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^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.

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.

Annex 4

Testing for Reactivity with Liquid Oxygen on Mechanical Impact

Approximately 0.5 g of the liquid or divided sample is placed into a sample cup (height = 10 mm; diameter = 30 mm), made of 0.01 mm copper foil. Liquid oxygen is poured into the cup over the sample which is then exposed to the mechanical impact of a plummet (mass = 76.5 kg). The drop height of the plummet can be varied. A steel anvil with a chrome/nickel steel plate supports the sample cup. The anvil, having a mass eight times of the plummet, is supported by four damping elements mounted on the steel frame of the test apparatus that rests on a concrete base.

A reaction of the sample with liquid oxygen is usually indicated by a flame and a more or less strong noise of an explosion. The impact energy, at which no reaction occurs, is determined in varying the drop height of the plummet. This result shall be confirmed in a series of ten consecutive tests under the same conditions. The tests are finished, if reactions can be observed at impact energies of 125 Nm or less (equivalent to a drop height of the plummet of 0.17 m or less). In this case, with regard to technical safety, the material is not suitable for liquid oxygen service.