BAM
Bundesanstalt für
Materialforschung
und -prüfung

12200 Berlin, Germany
P: +49 30 8104-0
F: +49 30 8104-7 2222

TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

BAM reference	16016326 E
Copy	1. copy of 2 copies
Customer	TEADIT International Produktions GmbH Rosenheimerstraße 10 6330 Kufstein Austria
Date of request	March 18, 2016
Receipt of signed contract	May 9, 2016
Reference	- - -
Test samples	Sealing material TEADIT 30SH, batch 24000179; BAM Order-No.: 2.1/53 148
Receipt of samples	May 9, 2016
Test date	May 20 to September 14, 2016
Test location	BAM – Division 2.1 „Gases, Gas Plants"; building no. 41, room 073 and room 120
Test procedure or requirement according to (in the current version at test time)	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 4.

TEST REPORT

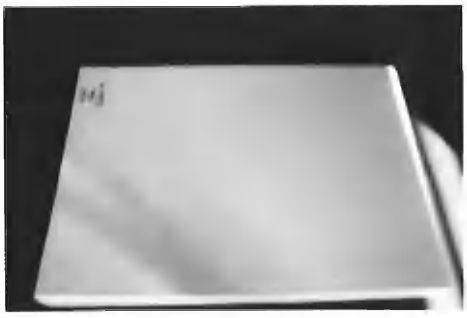
1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
„Testing and evaluating the nonmetallic material TEADIT 30SH, batch 24000179, for use as a sealing material in components for gaseous oxygen service at temperatures up to 200 °C and as a gasket in flange connections up to 30 bar.

1 Safety Data Sheet TEADIT 30SH
(5 Pages, Rev. 05/19112015)

8 Sheets of Sealing Material TEADIT 30SH, batch 24000179,
Dimension: 210 mm x 295 mm, Thickness 3,4 mm
Color of the sealing material: White



2 Applied Test Methods for Evaluating the Technical Safety

The product TEADIT 30SH is a nonmetallic material that shall be used as a sealing material in components for gaseous oxygen service at temperatures up to 200 °C or as a gasket in flange connections up to 30 bar. The following test method was applied:

2.1 Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

Generally, this test method is required if rapid oxygen pressure changes on the material cannot be safely excluded in usage.

2.2 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

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

The autogenous ignition temperature (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 100 °C below this AIT.

2.3 Testing 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 aging process.

2.4 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 inside 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 six discs were cut with a diameter of 140 mm from the samples. The discs were prepared as shown in figure 1.

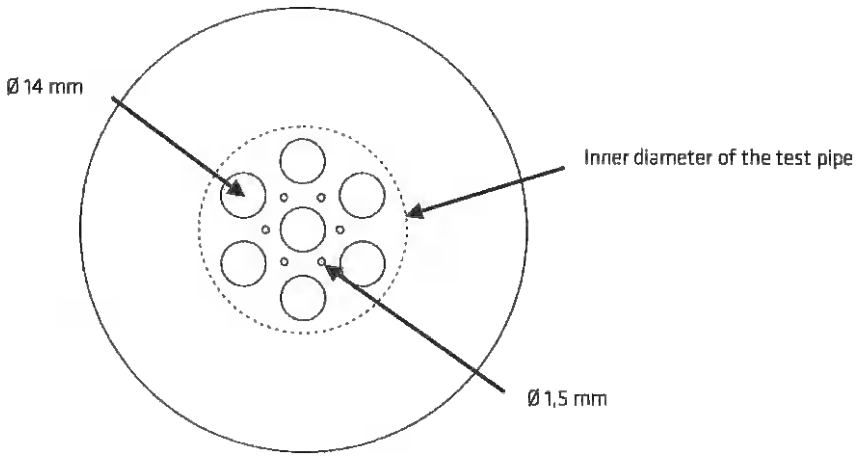


Figure 1: Preparation of the nonconductive material

For further tests, the material was cut into pieces of approximately 1 mm^3 up to 2 mm^3.

4 Tests

4.1 Ignition Sensitivity Testing to Gaseous Oxygen Impacts at 60 °C

The test method is described in annex 1. Based on the specified use conditions by the customer, the test was performed at 60 °C.

4.1.1 Assessment Criterion

According to DIN EN 1797 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010 „Cryogenic Vessels - Gas/Material Compatibility" the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

4.1.2 Results

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction
60	1	20	no reaction*
60	1	30	no reaction*
60	1	40	Ignition on 4. impact
60	1	30	Ignition on 1. impact
60	1	20	no reaction*
200	1	50	no reaction*
200	1	60	Ignition on 1. impact
200	1	50	Ignition on 5. impact
200	1	40	no reaction*
200	1	40	Ignition on 2. impact
200	1	30	Ignition on 3. impact
200	1	20	no reaction*
200	1	20	Ignition on 3. impact
200	1	10	no reaction*
200	1	10	no reaction*

* Within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of the sample with oxygen could be observed at following conditions:

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]
60	1	20
200	1	10

4.2 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

The test method is described in annex 2. Based on the test results of the oxygen impact test at 60 °C, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 20 bar.

4.2.1 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.

4.2.2 Results

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	8	21	446
2	8	21	450
3	8	21	446
4	8	22	453
5	8	21	444

Five tests resulted in the following mean AIT and its corresponding standard deviation:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
21	448	± 4

4.3 Aging Behavior

The test method is described in annex 3. In general, the aging test is carried out at the maximum operating pressure and at an elevated temperature, which is 25 °C above the maximum operating temperature. In this case, the aging test was carried out at 20 bar and at 225 °C.

4.3.1 Assessment Criterion

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 samples 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.

4.3.2 Results

4.3.2.1 Change of Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	225	20	± 0.0

After aging, the mass of the test sample did not change and the test sample was apparently unchanged.

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

The test method is described in annex 2. The AIT test of the aged material was performed at same conditions as described in chapter 4.2 of the non-aged material.

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	8	21	459
2	8	21	443
3	8	21	442
4	8	21	448
5	8	21	448

Five tests resulted in the following mean AIT and its corresponding standard deviation:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
21	448	± 7

4.4 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 4. Based on the specified use conditions by the customer the flange test was performed at a final oxygen pressure of approximately 30 bar and 200 °C.

4.4.1 Assessment Criterion

If 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 flanges and the flange connection is still gas tight there are no objections with regard to technical safety to use the gasket under the conditions tested. Such a positive result has to be confirmed in four additional tests.

If the gasket 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 right from the beginning.

If, however, the flange connection becomes un-tight during a test, e. g., because of softening or burning of the gasket, 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.

4.4.2 Results

Test Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	200	30	Parts of the gasket burn approximately 2,9 mm between the flanges. The flange connection remains gas-tight. The fire is not transmitted to the steel.
2	200	20	Only those parts of the gasket burn that project into the pipe. The flange connection remains gas-tight.
3	200	20	same behavior as in test no. 2
4	200	20	same behavior as in test no. 2
5	200	20	same behavior as in test no. 2
6	200	20	same behavior as in test no. 2

In five tests at 20 bar oxygen pressure and 200 °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. After the tests the sealing part of the gaskets has a thickness of approximately 1.1 mm.

5 Summary and Evaluation

It is intended to use the product TEADIT 30SH as a sealing material in components for gaseous oxygen service and as a gasket material in flange connections.

Ignition sensitivity testing of the material showed that no reactions could be detected at temperatures of 60 °C and a final oxygen pressure of 20 bar and at 200 °C and a final pressure of 10 bar.

In five tests with an initial oxygen pressure of p_I = 8 bar, an AIT of 448 °C was determined with a standard deviation of ± 4 °C. The oxygen pressure p_F at ignition is approximately 21 bar.

After aging of the test sample at 20 bar oxygen pressure and 225 °C, the mass of the test sample did not change and the test sample was apparently unchanged.

Based on the test results, there are no objections with regard to technical safety, to use the sealing material TEADIT 30SH, batch 24000179, in components for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	20
> 60 up to 200	10

Flange testing of the gasket material showed that in five tests at 20 bar oxygen pressure and 200 °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.

Additionally, there are also no objections with regard to technical safety to use the gasket material TEADIT 30SH, batch 24000179, with a maximum thickness of 3.4 mm in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
200	20

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

This evaluation does not cover the use of the sealing material TEADIT 30SH, batch 24000179, for liquid oxygen service. For this case, a particular test for reactivity with liquid oxygen needs to be carried out.

6 Comments

This safety evaluation considers the fact, that rapid oxygen pressure changes – so called oxygen pressure surges – cannot be safely excluded on the material in usage.

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

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 only be used for gaseous 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

November 2, 2016

Division 2.1 "Gases, Gas Plants"

By order

Dipl.-Ing. Peter Hartwig

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

Annex 1

Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

Approximately 0.2 g to 0.5 g of the pasty or divided solid sample is placed into a heatable steel tube, 15 cm^3 in volume. In case of liquids to be tested, ceramic fibre, soaked with the sample, is used. The sample tube is connected by a 750 mm long pipe (internal diameter 14 mm) and a pneumatically operated quick opening valve to a high-pressure oxygen accumulator.

A heater allows to set the sample tube to the test temperature t_a After the tube and pipe are at test pressure p_I, the quick opening valve is opened and preheated oxygen of 60 °C and of pressure p_F flows abruptly into the pipe and tube. In this way, the oxygen in the tube and in the pipe is almost adiabatically compressed from pressure p_I to p_F and heated. If there is a reaction of the sample with oxygen, indicated by a steep temperature rise in the tube, further tests with a new sample are performed at a lower pressure ratio p_F/p_I. If, however, no reaction of the sample with oxygen can be detected after a waiting period of 30 seconds, the tube is de-pressurized and the test is repeated (up to four times) until a reaction takes place. This means, each test series consists of a maximum of five single tests with the same material under the same conditions. If no reaction can be observed, even after the fifth single test of a test series, testing is continued with new samples at greater pressure ratios p_F/p_I, until finally that pressure ratio is determined, at which no reaction can be observed within a test series of five single tests.If the repetition of that test series with a new sample shows the same result, the test can be finished or continued at a different test temperature t_a.

Annex 2

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 3

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 4

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.