BAM
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TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

Reference Number	16037461 E
Copy	1. copy of 2 copies
Customer	TEADIT International Produktions GmbH Rosenheimerstraße 10 6330 Kufstein Austria
Date of Request	August 8, 2016
Reference	-
Receipt of Award of Contract	September 16, 2016
Test Samples	Joint sealant TEADIT 24B with adhesive, Undisclosed batch; BAM Order-No.: 2.1/53 266
Receipt of Samples	September 16, 2016
Test Date	November 22, 2016 to February 10, 2017
Test Location	BAM – Division 2.1 „Gases, Gas Plants"; building no. 41
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 7 and annex 1 to 3.

2015-06 / 2015-09-17

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
„Testing and evaluating the joint sealant TEADIT 24B with adhesive, undisclosed batch, for gaseous oxygen service at 100 bar and 100 °C."

1 Safety Data Sheet
TEADIT 24B
(5 Pages, Rev. 06/19112015)

2 m Sealing tape TEADIT 24B with adhesive, undisclosed batch,
Dimensions: Width 10 mm, Thickness 3 mm
Color of the sealing material: White

2 Applied Test Methods for Evaluating the Technical Safety

Tests on ignition sensitivity to gaseous oxygen impacts were not carried out because oxygen pressure impacts can be safely excluded in the intended service according to the information by the customer.

The nonmetallic material is intended for use as a sealing material in flange connections for gaseous oxygen service at 100 bar and temperatures up to 100 °C.

The following test methods were applied:

2.1 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

Usually, this test method is required if the material is for service 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.

For sealing materials in flange connections, the safety margin between AIT and maximum use temperature is only 50 °C because of the particularly mounting situation.

2.2 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 processes.

2.3 Testing of Sealing Materials for Flanges in High Pressure Oxygen

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

3 Preparation of Samples

To test the sealing tape with adhesive it was prepared as shown in figure 1.

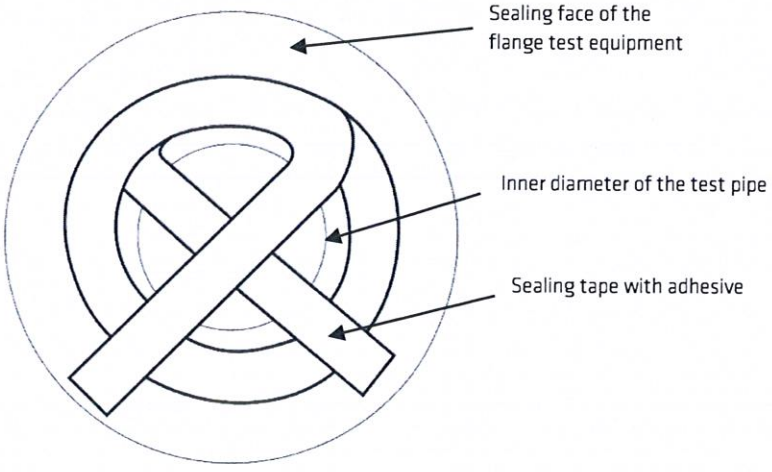

Figure 1: Preparation of the sealing tape with adhesive

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

4 Tests

4.1 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

The test method is described in annex 1. It was performed with a final oxygen pressure of approximately 100 bar according to the requirement 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	70	103	155
2	70	102	150
3	70	106	170
4	70	106	165
5	70	103	156

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]
104	159	± 8

4.2 Aging Behavior

The test method is described in annex 2. 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 100 bar and at 125 °C.

4.2.1 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 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 of Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	125	100	- 0,3

After aging, the color of the adhesive was changed to brown although the test sample was apparently unchanged. The sample lost 0.3 % 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 of the non-aged material.

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	70	114	198
2	70	111	182
3	70	115	204
4	70	110	181
5	70	113	190

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]
113	191	± 10

4.3 Testing of Sealing Materials for Flanges in High Pressure Oxygen

The test method is described in annex 3. Based on the specified use conditions by the customer the flange test was performed at a final oxygen pressure of approximately 100 bar and 100 °C.

4.3.1 Assessment Criterion

If only those parts of the sealing material burn that project into the pipe and the fire is not transmitted to the flanges and if the sealing material 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 sealing material under the conditions tested. Such a positive result has to be confirmed in four additional tests.

If the sealing material 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 sealing material, 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.

4.3.2 Results

Test Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	100	100	Only those parts of the sealing material burn that project into the pipe. The flange connection remains gas-tight.
2	100	100	same behavior as in test no. 1
3	100	100	same behavior as in test no. 1
4	100	100	same behavior as in test no. 1
5	100	100	same behavior as in test no. 1

In five tests at 100 bar oxygen pressure and 100 °C, only those parts of the sample burn that project into the pipe. The fire is neither transmitted to the steel nor does the sample burn between the flanges. The flange remains gas-tight. After the tests the sealing part of the sample has a thickness of approximately 1.5 mm.

5 Summary and Evaluation

It is intended to use the product TEADIT 24B with adhesive as a sealing material in flange connections for gaseous oxygen service.

After aging of the test sample at 100 bar oxygen pressure and 125 °C, the sealing material proved to be sufficient aging resistant. Based on the change of the adhesives color, it proved to be insufficient aging resistant at these conditions. The sample lost 0.3 % in mass. The noticed difference of the adhesiv´s color has not influence of the application in oxygen, but it can influence the practical application of the sealing material.

Based on the test results and the aging behavior of the adhesive, there are no objections regarding to technical safety to use the sealing material TEADIT 24B with adhesive, undisclosed batch, with a maximum thickness of 3. mm in flange connections made of copper, copper alloys or steel at following conditions:

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

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 24B, undisclosed batch, 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 –can be safely excluded 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 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

March 27, 2017

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

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.