

Report

on Testing a Gasket Material for Reactivity with Oxygen

Reference Number	2-1244/2012 I E
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Customer	Frenzelit-Werke GmbH Frankenhammer 7 95460 Bad Berneck Germany
Order Date	April 24, 2012
Reference	EMP / BWI
Receipt of Order	May 16, 2012
Test Samples	Gasket material novatec® PREMIUM XP for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service and for liquid oxygen service. BAM-Order No. 2.1/51 093
Receipt of Samples	May 15, 2012
Test Date	July 25, 2012 to November 27, 2012
Test Location	BAM – Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073
Test Procedure or Requirement According to	DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" ISO 21010: 2004-07 „Cryogenic Vessels - Gas/Material Compatibility" Annex of pamphlet M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen by BAM Federal Institute for Material Research and Testing.", by Berufsgenossenschaft Rohstoffe und chemische Industrie, Edition: September 2011; Rule BGR 500 "Betreiben von Arbeitsmitteln" part 2, chapter 2.32 "Betreiben von Sauerstoffanlagen", paragraph 3.17 "Lubricants and sealing materials", Edition: April 2008.

All pressures of this report are excess pressures.
This test report consists of page 1 to 5 and annex 1 to 4.

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In case a German version of the test report is available, exclusively the German version is binding.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test Application

"Evaluate the compatibility of the gasket material novatech® PREMIUM XP for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at temperatures greater than 60 °C and 130 bar and for liquid oxygen service.

10 Disks of the gasket material novatec® PREMIUM XP

Diameter 140 mm; Thickness 3 mm

With Imprint: novatech® PREMIUM XP

Colour: Blue

2 Test Methods

To evaluate the compatibility of the gasket material novatec® PREMIUM XP for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance in high pressure oxygen with an re-investigation of the aged material, and the "Flange Test" were carried out at an oxygen pressure of 130 bar.

The compatibility of the material with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Autogenous Ignition Temperature (AIT)

The test method is described in annex 1.

Results:

Test No.	Initial Oxygen Pressure p_i [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	92	136	163
2	92	132	150
3	92	135	157
4	92	133	153
5	92	133	149

In five tests with an initial oxygen pressure of $p_i = 92$ bar, an AIT of 154 °C was determined with a standard deviation of ± 6 °C. The oxygen pressure p_F at ignition is approximately 134 bar.

3.2 Artificial Aging

The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	130	130	-4.5

After aging of the test sample at 130 bar oxygen pressure and 130 °C the test sample was highly embrittled. The sample lost 4.5 % in mass.

3.2.1 AIT after Artificial Aging

The test method is described in annex 1.

Results:

Number of Tests	Initial Oxygen Pressure p_i [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	92	188	323
2	92	189	331
3	92	194	337
4	92	192	332
5	92	189	323

In five tests with an initial oxygen pressure of $p_i = 92$ bar, an AIT of 329 °C was determined with a standard deviation of ± 6 °C. The final oxygen pressure p_F at ignition is approximately 191 bar.

3.3 Flange Test

The test method is described in annex 3.

Results:

Number of Tests	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	105	130	Only those parts of the gasket ¹ burn that project into the pipe.
2	105	130	same behavior as in test no. 1
3	105	130	same behavior as in test no. 1
4	105	130	same behavior as in test no. 1
5	105	130	same behavior as in test no. 1

In five tests at 130 bar oxygen pressure and 105 °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.

3.4 Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

Results:

Test No.	Drop Heights [m]	Impact Energy [Nm]	Reaction
1	0.67	500	reaction
2	0.50	375	no reaction
3	0.50	375	no reaction
4	0.50	375	reaction
5	0.33	250	no reaction
6	0.33	250	no reaction
7	0.33	250	reaction
8	0.17	125	no reaction
9	0.17	125	no reaction
10	0.17	125	no reaction
11	0.17	125	no reaction
12	0.17	125	no reaction
13	0.17	125	no reaction
14	0.17	125	no reaction
15	0.17	125	no reaction
16	0.17	125	no reaction
17	0.17	125	no reaction

At drop heights of 0.67 m to 0.33 m (impact energy 500 Nm to 250 Nm), severe reactions of the sample with liquid oxygen occurred.

No reaction of the sample with liquid oxygen could be detected at a drop height of 0.17 m (Impact energy 125 Nm), in ten separate tests.

4 Summary and Evaluation

The product novatec® PREMIUM XP had already been tested and evaluated in 2005 and 2006 as a gasket material for use in valves and fittings or other components for gaseous oxygen service under reference numbers 2005, Tgb.-Nr.: II-1492/2005 E, und 2006, Tgb.-Nr.: II-304/2006.

The tests have shown that the autogenous ignition temperature of the gasket material novatec® PREMIUM XP is 154 °C at 134 bar oxygen pressure. The standard deviation of the AIT is ± 6 °C.

At a temperature of 130 °C and an oxygen pressure of 130 bar, the gasket material novatec® PREMIUM XP proved to be insufficient aging resistant. The test sample was highly brittle and the sample lost 4,5 % in mass.

Therefore the gasket material novatec® PREMIUM XP may only be used for gaseous oxygen service, if dynamic stresses can safely be excluded. In addition, the unfavorable aging behaviour may influence the practical application of the material.

The tests have shown that the autogenous ignition temperature of the aged gasket material novatec® PREMIUM XP is 329 °C at 191 bar oxygen pressure. The standard deviation of the AIT is ± 6 °C.

This shows, that the AIT of the aged sample is significantly greater than the AIT of the non-aged sample within the precision of measurement.

On basis of the test results and the result of the AIT of the non-aged material, there are no objections with regard to technical safety to use the gasket material novatec® PREMIUM XP in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature	Maximum Oxygen Pressure
up to 100 °C	up to 130 bar

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

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", described in annex 4, there are no objections with regard to technical safety to use the gasket material novatec® PREMIUM XP in piping, valves and fittings or other components for liquid oxygen service. In this case, a limitation to a particular pressure range is not necessary as compression of liquid oxygen causes no significant change in concentration and therefore has no considerable influence on the reactivity of the gasket material.

5 **Comments**

The test results refer exclusively to the batch of the tested material.

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.

It shall be clear that the product may only be used for gaseous oxygen service and liquid 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.

**BAM Federal Institute for Materials Research and Testing
12200 Berlin, December 3, 2012**

Division 2.1

"Gases, Gas Plants"



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Study Director "Safe Handling of Oxygen"

Copies:

1. Copy: Frenzelit-Werke 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 cm³ in volume) with a chrome/nickel lining. Liquid samples are applied onto ceramic fiber.

The autoclave is pressurized to the desired pressure p_a 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 pressure. The oxygen pressure on ignition p_e is calculated.

It is important to know the oxygen pressure p_e , 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.