

Report

on Testing a Gasket Material for Reactivity with Oxygen

Reference Number	2-1379/2011 I E
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Customer	GARLOCK GmbH Falkenweg 1 41468 Neuss
Order Date	June 1, 2011
Receipt of Order	June 9, 2011
Test Samples	Gasket material GYLON STYLE 3504 blue for use in flanged connections in piping, valves and fittings or other components for gaseous and liquid oxygen service. BAM-Order No. 2.1/50 633
Receipt of Samples	June 7, 2011
Test Date	September 8, 2011 to February 25, 2012
Test Location	BAM – Working Group “Safe Handling of Oxygen”; building no. 41, room no. 073 and no. 120
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.

TEST REPORT

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test Application
- 1 Safety Data Sheet
- 1 Material Data Sheet
- 15 Disks GYLON STYLE 3504 blue
 - Diameter 140 mm; Thickness 2 mm
 - Colour: Light Blue

2 Test Methods

In 2000 the material has already been tested and evaluated as a gasket material for use in flanged connections in piping, valves and fittings or other components for gaseous and/or liquid oxygen service under reference number Tgb.-Nr. II-2421/2000. In this case the tests to determine the compatibility for gaseous oxygen service at maximum operating conditions of 260 °C and 55 bar were carried out.

To test and evaluate the compatibility of the gasket material GYLON STYLE 3504 blue for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at temperatures up to 260 °C and 55 bar oxygen pressure a flange test at 260 °C and 55 bar, a determination of the autogenous ignition temperature (AIT) and an investigation of the aging resistance in high pressure oxygen were carried out.

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	22	56	451
2	22	56	455
3	22	56	452
4	22	56	455
5	22	56	454

In five tests with an initial oxygen pressure of $p_i = 22$ bar, an AIT of 453 °C was determined with a standard deviation of ± 2 °C. The final oxygen pressure p_f is approximately 56 bar.

3.2 Artificial Aging

The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	285	55	-0,1

After aging of the test sample at 55 bar oxygen pressure and 285 °C, the test sample was apparently unchanged. The sample lost 0,1 % 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	22	56	457
2	22	56	452
3	22	56	454
4	22	56	457
5	22	56	455

In five tests with an initial oxygen pressure of $p_i = 22$ bar, an AIT of 455 °C was determined with a standard deviation of ± 2 °C. The final oxygen pressure p_f is approximately 56 bar.

This shows, that the AIT of the aged sample is almost unchanged compared to the AIT of the non-aged sample within the precision of measurement.

3.3 Flange Test

The test method is described in annex 3.

Results:

Number of Tests	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	55	260	The gasket burns 4 mm to 6 mm between the flanges. Based on this test result the test conditions were changed in accordance with the results made in 2000.
2	25	200	Only those parts of the gasket burn that project into the pipe.
3	25	200	same behavior as in test no. 2
4	25	200	same behavior as in test no. 2
5	25	200	same behavior as in test no. 2
6	25	200	same behavior as in test no. 2

In five tests at 25 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.

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	1,00	750	ignition on impact
2	0,83	625	ignition on impact
3	0,67	500	no reaction
4	0,67	500	ignition on impact
5	0,50	375	no reaction
6	0,50	375	no reaction
7	0,50	375	no reaction
8	0,50	375	no reaction
9	0,50	375	no reaction
10	0,50	375	no reaction
11	0,50	375	no reaction
12	0,50	375	no reaction
13	0,50	375	no reaction
14	0,50	375	no reaction

At a drop height of 0,50m (impact energy 375 Nm) no reaction of the test sample with liquid oxygen could be detected in ten separate tests.

4 Summary and Evaluation

The tests have shown that the autogenous ignition temperature of the gasket material GYLON STYLE 3504 blue is 453 °C at 56 bar oxygen pressure. The standard deviation of the AIT is ± 2 °C.

At a temperature of 285 °C and an oxygen pressure of 55 bar, the gasket material GYLON STYLE 3504 blue proved to be sufficient aging resistant. The sample lost 0,1 % in mass.

On basis of those test results and the results of the flange testing, there are no objections with regard to technical safety to use the gasket material GYLON STYLE 3504 blue in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature	Maximum Oxygen Pressure
up to 200 °C	up to 25 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 GYLON STYLE 3504 blue in 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 which would include e. g. the monitoring of the production.

It shall be clear that the product may only be used for gaseous 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, 13th April 2012**

**Division 2.1
"Gases, Gas Plants"**



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- Working Group "Safe Handling of Oxygen" -

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.

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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.

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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.



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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.