



TEST REPORT

BAM reference	22043758-E
Copy	1 st copy of 2
Customer	Rich. Klinger Dichtungstechnik GmbH & Co. KG Am Kanal 8 - 10 2352 Gumpoldskirchen Austria
Date of Request	October 20, 2022
Your Reference	-
Receipt of Contract	November 23, 2022
Test Samples	PTFE-based gasket material Klinger®top-chem 2006, batch 06636
Receipt of Samples	October 14, 2022
Test Date	November 23, 2022, to March 6, 2023
Test Location	Division 2.1 "Safety of Energy Carriers"; 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"

This test report consists of page 1 to 8 and enclosures 1 to 3

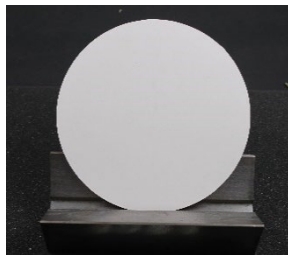
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The German version is legally binding, except an English version is issued exclusively.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test application
Safety-related investigation on PTFE-based gasket material
Klinger®top-chem 2006, batch 06636, for use in gaseous oxygen service at
temperatures up to 200 °C and pressures up to 130 bars
- 1 Sicherheitsdatenblatt Klinger®top-chem 2006
(6 Seiten, Rich. Klinger Dichtungstechnik GmbH & Co. KG,
Erstelldatum: 20.01.2011)
- 1 Stellungnahme zur SVHC Candidate List der REACH Verordnung
Klinger®top-chem
(1 Seite, Klinger Dichtungstechnik, Erstelldatum: 08.08.2022)
- 1 Completely filled Customer Master Data Sheet (CMDS) (October 20, 2022)
- 16 Disks of PTFE-based gasket material Klinger®top-chem 2006, batch 06636
Dimensions: Ø 140 mm, Thickness 2 mm
Color: White



2 Applied Test Methods

The PTFE-based gasket material Klinger®top-chem 2006, batch 06636, shall be used for gaseous oxygen service at temperatures up to 200 °C and at pressures up to 130 bars.

Reactivity testing of the PTFE-based gasket material Klinger®top-chem 2006, batch 06636, to rapid oxygen pressure changes, so-called oxygen pressure impacts, was not carried. According to the information by the customer and in the intended application, 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. Therefore, it is relevant for the maximum use temperature that is generally set 50 °C below this AIT.

2.2 Testing for Aging Resistance 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.

3 Sampling

The material sample used for the investigation was provided by the customer.

3.1 Preparation of Samples

For testing the “Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen”, the PTFE-based gasket material Klinger®top-chem 2006, batch 06636, was cut into parts of ca. 1 to 2 mm in edge length and was used in this form.

For testing the “Aging Resistance in High Pressure Oxygen”, the PTFE-based gasket material Klinger®top-chem 2006, batch 06636, was cut into parts of ca. 40 to 20 mm in edge length and was used in this form.

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

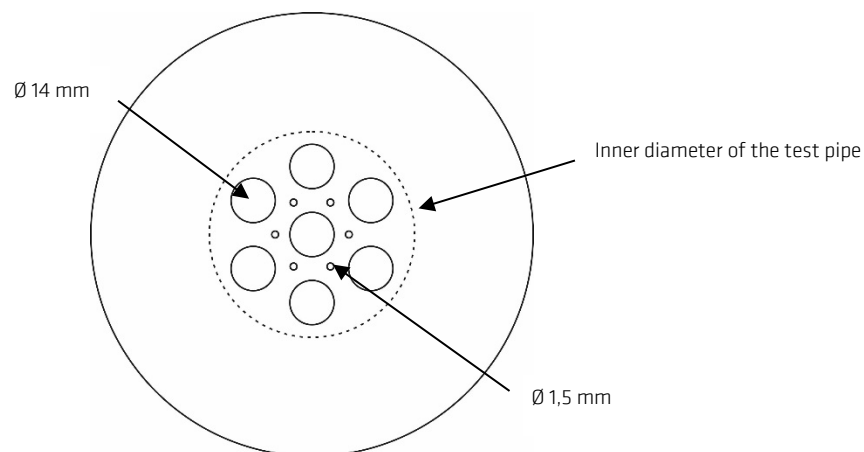


Figure 1: Preparation of the nonconductive flat gasket material

4 Tests

4.1 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

The test method is described in annex 1.

Based on the intended use conditions, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 130 bars.

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.	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	130	463
2	133	462
3	131	461
4	134	467
5	133	464

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

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
132	463	± 2

4.2 Testing for Aging Resistance 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 at an elevated temperature, that is 25 °C above the maximum operating temperature. Consequently, the test temperature was 225 °C. Based on the intended maximum use pressure of the material, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 130 bars.

4.2.1 Assessment Criteria

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 Testing for Change in Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	225	130	± 0.0

After aging, the test sample was apparently unchanged and has no loss 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 at same conditions as described in chapter 4.1.

Test No.	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	131	460
2	133	462
3	134	466
4	134	466
5	134	469

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

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
133	465	± 4

4.3 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 3.

Based on the intended use conditions, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 130 bars and at a temperature of 200 °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

During the filling process, the material suddenly ignited at an oxygen pressure of 125 bars and at a temperature of 200 °C before the test has even started. The material was blown out of the flange connection. After consultation with the customer, BAM was informed by email that the flange test should be repeated at an oxygen pressure of 100 bars and at a temperature of 200 °C.

Test Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	200	100	All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas tight.
2	200	100	Same behavior as in test no. 1
3	200	100	Same behavior as in test no. 1
4	200	100	Same behavior as in test no. 1
5	200	100	Same behavior as in test no. 1

In five tests at 200 °C and an oxygen pressure of 100 bars, 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. After the tests, the tested samples had a thickness of 2 mm.

5 Summary of the Test Results

At a mean final oxygen pressure p_F of 132 bars, the test sample has a mean autogenous ignition temperature of 463 °C with a standard deviation of ± 2 °C.

The material proved to be aging resistant at 225 °C and at 130 bars oxygen pressure.

Investigation of the burning behavior of disks of the gasket material in a standard flange showed that the material reacted at a temperature of 200 °C and an oxygen pressure of 125 bars during the filling process.

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

6 Measurement uncertainty

The tests are carried out in accordance with the standards or guidelines indicated on the cover sheet of this report. Thereafter, the temperature measurement should have a maximum deviation of ± 2 K and the pressure measurement should have a maximum deviation of ± 2 bar.

For the test in chapter 4.1, the extended uncertainty is 0.7 K (according to the calibration protocol from January 23, 2023) for the temperature measuring system, and the uncertainty is 0.3 bar (according to the calibration protocol from January 25, 2023) for the used pressure measuring system.

For the test in chapter 4.2, the extended uncertainty is 1.0 K (according to the calibration protocol from January 14, 2021) for the temperature measuring system, and the uncertainty is 0.3 bars (according to the calibration protocol from January 14, 2021) for the used pressure measuring system.

For the test in chapter 4.3, the extended uncertainty is 2.8 K (according to the calibration protocol from February 27, 2023) for the temperature measuring system, and the uncertainty is 0.7 bars (according to the calibration protocol from September 8, 2022) for the used pressure measuring system.

7 Statements of conformity

The tests are carried out in accordance with the standards or guidelines, stated on the cover sheet of this report. Deviating or supplementary test criteria are described in the respective subchapter "Assessment Criterion" in Chapter 4 "Tests".

8 Opinion and Interpretation

The PTFE-based gasket material Klinger®top-chem 2006, batch 06636, was originally intended to be used in gaseous oxygen at temperatures up to 200 °C and at pressures up to 130 bars. Due to the failed flange test, the customer lowered the intended operating conditions to 100 bars and 200 °C.

On basis of the test results, the requirements for sealing materials, described in the code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and based on the assessment criteria described in this test report, there are no objections regarding technical safety, to use the PTFE-based gasket material Klinger®top-chem 2006, batch 06636, for gaseous oxygen service at following use conditions only:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
200	100

9 Comments

This safety-related investigation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges – on the material can be safely excluded in usage. In addition, the safety related investigation considers the fact, that the material shall be used in gaseous oxygen service at temperatures greater than 60 °C.

The content of the test report refers exclusively to the test sample of the the PTFE-based gasket material Klinger®top-chem 2006, batch 06636.

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 JA1102309. 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 the specified purpose in 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

April 4, 2023

Division 2.1 “Safety of Energy Carriers”

by order

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