BAM
Bundesanstalt für
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und -prüfung

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TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

Reference Number	16017264 I E
Copy	1. copy of 2 copies
Customer	Chemours International Operations Sàrl Chemin du Pavillon 2, P.O Box 50 1218 Le Grand-Saconnex, Geneva Switzerland
Date of Request	April 7, 2016
Receipt of Signed Contract	June 21, 2016
Reference	- - -
Test Samples	Lubricant Krytox™ NRT 8908, batch 011516-1; BAM Order-No.: 2.1/53 161
Receipt of Samples	June 20, 2016
Test Date	June 23 to September 13, 2016
Test Location	BAM – Division 2.1 „Gases, Gas Plants"; building no. 41, room 073 and 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 7 and annexes 1 to 3.

2015-06 / 2015-09-17

2015-06

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1	Test application „Testing and evaluating Krytox™ NRT 8908, batch 011516-1, for use as lubricant in gaseous and liquid oxygen service at specified operating conditions"
1	Safety Data Sheet Krytox™ NRT 8908 (11 pages, version 4.0, date of issue: 20.10.2015)
100 g	Paste-like lubricant Krytox™ NRT 8908 in a white plastic cup; Color of the lubricant: White

2 Applied Test Methods for Evaluating the Technical Safety

The product Krytox™ NRT 8908 is a lubricant that shall be used for gaseous oxygen service at temperatures up to 200 °C and for liquid oxygen service. The following test methods were 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 ignition temperature.

2.3 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

Generally, this test method is required if direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage.

3 Preparation of Samples

Prior to testing, the lubricant was stirred with a spatula for homogenization.

4 Tests

4.1 Ignition Sensitivity Testing to Gaseous Oxygen Impacts

The test method is described in annex 1. Based on the specified use conditions by the customer, the test was performed at 60 °C, at 100 °C, at 150 °C, and at 200 °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.

If the sample exhibits a change of color, or of consistency after testing, this is also considered as a positive reaction by BAM for safety reasons, even if there is no temperature rise detectable 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	360	Ignition on 1. impact
60	1	340	no reaction*
60	1	350	no reaction*
60	1	350	Ignition on 2. impact
60	1	340	no reaction*

* Within a series of five consecutive impacts

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction
100	1	340	Ignition on 5. impact
100	1	330	Ignition on 1. impact
100	1	320	Ignition on 2. impact
100	1	300	Ignition on 1. impact
100	1	280	Ignition on 1. impact
100	1	260	Ignition on 1. impact
100	1	240	Ignition on 1. impact
100	1	210	no reaction*
100	1	220	Ignition on 5. impact
100	1	210	no reaction*
150	1	210	no reaction*
150	1	210	no reaction*
200	1	210	no reaction*
200	1	210	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	340
100	1	210
150	1	210
200	1	210

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, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 250 °bar. This equals the maximum working pressure of the test equipment.

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	100	264	> 500
2	100	263	> 500
3	100	264	> 500
4	100	264	> 500
5	100	260	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of p_I = 100 bar. The final oxygen pressure p_F was approximately 263 bar.

Testing the autogenous ignition temperature can only be performed up to 500 °C. This equals the maximum working temperature of the test equipment.

4.3 Reactivity Testing with Liquid Oxygen on Mechanical Impact

The test method is described in annex 3.

4.3.1 Assessment Criterion

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", a nonmetallic material is not compatible with liquid oxygen, if reactions occur at a drop height of 0.17 m (impact energy 125 Nm) or less.

4.3.2 Results

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	0.83	625	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	no reaction
5	1.00	750	no reaction
6	1.00	750	no reaction
7	1.00	750	no reaction
8	1.00	750	no reaction
9	1.00	750	no reaction
10	1.00	750	no reaction
11	1.00	750	no reaction

At a drop height of 1.00 m (impact energy 750 Nm), in ten separate tests, no reaction of the sample with liquid oxygen could be detected.

5 Summary and Evaluation

It is intended to use the product as a lubricant for gaseous and for liquid oxygen service.

Ignition sensitivity testing of the material showed that no reactions could be detected at a temperature of 60 °C at a final oxygen pressure of 340 bar. Ignition sensitivity testing of the material also showed that no reactions could be detected at temperatures of 100 °C, 150 °C, and 200 °C at a final oxygen pressure of 210 bar.

In determining the AIT, up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with final oxygen pressures p_F = 263 bar.

Based on the test results, there are no objections with regard to technical safety, to use the lubricant Krytox™ NRT 8908, batch 011516-1, for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	340
> 60 up to 200	210

Based on the test results, there are also no objections with regard to technical safety to use Krytox™ NRT 8908, batch 011516-1, as a lubricant 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 material.

6 Comments

This safety evaluation considers the facts, that on the one hand rapid oxygen pressure changes - so-called oxygen pressure surges – and on the other hand direct contact of the material with liquid oxygen and mechanical impacts cannot 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 be used for gaseous and for 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.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

October 5, 2016

Division 2.1 "Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list:	1. copy:	Chemours International Operations Sàrl
	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.

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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 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.