

# Report

## on Testing a Lubricant for Reactivity with Oxygen

Reference Number

2-2075/2012 E

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Customer

**IKV** Tribologie

ZI de la gare - BP 31 26260 SAINT DONAT

**FRANKREICH** 

**Order Date** 

July 23, 2012

Reference

SK/20120723-R1

Receipt of Order

July 31, 2012

**Test Samples** 

Lubricant IKV-TRIBOFLON MYA 242 FG, Batch No. 122812, for use in piping, valves and fittings or other components for gaseous oxygen service at temperatures

greater than 60 °C.

BAM-Order-No.: 2.1/51 188

Receipt of Samples

July 25, 2012

**Test Date** 

August 21, 2012 to August 27, 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 2.

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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
- 1 Safety Data Sheet
- 1 Material Data Sheet
- 50 g Paste Lubricant IKV-TRIBOFLON MYA 242 FG, Batch No. 122812 Colour: White

#### 2 Test Methods

To test and evaluate the compatibility of the Lubricant IKV-TRIBOFLON MYA 242 FG, Batch No. 122812, for gaseous oxygen service at temperatures greater than 60 °C the ignition sensitivity to gaseous oxygen impacts and a determination of the autogenous ignition temperature (AIT) were carried out.

# 3 Results

# 3.1 Autogenous Ignition Temperature (AIT)

The test method is described in annex 1.

#### Results:

Test No.	Initial	Final	AIT
	Oxygen	Oxygen	[°C]
	Pressure	Pressure	
	p <sub>i</sub> [bar]	p <sub>F</sub> (bar)	
1	44	93	346
2	44	92	344
3	44	92	338
4	44	94	349
5	44	94	351

In five tests with an initial oxygen pressure of  $p_I$  = 44 bar, an AIT of 346 °C was determined with a standard deviation of  $\pm$  5 °C. The oxygen pressure  $p_F$  at ignition is approximately 93 bar.

# 3.2 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 2.

Results:

Sample	Initial	Final	Reaction	
Temperature	Oxygen	Oxygen	on	
•	Pressure	Pressure		
t <sub>a</sub> [°C]			Impact	
60	p <sub>i</sub> [bar]	p <sub>F</sub> [bar] 150	ignition on	
60	1	150	1. impact	
60	1	140	ignition on	
60	•	140	1. impact	
60	4	130	ignition on	
60	1	130	•	
00	4	400	1. impact	
60	1	120	ignition on	
00	4	440	1. impact	
60	1	110	ignition on	
00	4	400	1. impact	
60	1	100	ignition on	
00	4	00	1. impact	
60	1	90	no reaction*	
60	1	90	no reaction*	
400	4	00	iamitian an	
100	1	90	ignition on	
400	4	00	1. impact	
100	1	80	ignition on	
400	4	70	1. impact	
100	1	70	ignition on	
400	4	60	2. impact	
100	1	60	ignition on	
400	4	ΕΛ	1. impact	
100	1	50 50	no reaction*	
100	1	50	no reaction*	
450	4	FO	ionition on	
150	1	50	ignition on	
150	4	40	1. impact	
150 450	1 1	40	no reaction* no reaction*	
150	ı	40	no reaction	
200	4	40	no reaction*	
200	1 1	40 40	no reaction*	
200	i	40	no reaction	
250	1	40	no reaction*	
250	ı	40		
250	1	30	-	
	_			
250 250 250 250	1 1 1 1	40 40 30 30	no reaction* ignition on 5. impact no reaction* no reaction*	

<sup>\*</sup> 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 the following test conditions:

Sample	Final	
Temperature	Oxygen	
t <sub>a</sub> [°C]	Pressure	
	p <sub>F</sub> [bar]	
60	90	
100	50	
150	40	
200	40	
250	30	

## **4** Summary and Evaluation

The tests have shown that the autogenous ignition temperature of the Lubricant IKV-TRIBOFLON MYA 242 FG, Batch No. 122812, is 346  $^{\circ}$ C at 93 bar oxygen pressure. The standard deviation of the AIT is  $\pm$  5  $^{\circ}$ C.

According to DIN EN 1797: 2002-02 "Kryo-Behälter - Verträglichkeit von Gas/Werkstoffen" and to ISO 21010: 2004-07 "Cryogenic Vessels - Gas/Material Compatibility" the criterion for a positive reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use the Lubricant IKV-TRIBOFLON MYA 242 FG, Batch No. 122812, in piping, valves and fittings, or other components for gaseous oxygen service at following operating conditions:

Maximum Temperature	Maximum Oxygen Pressure	
up to 60 °C	up to 90 bar	
>60 °C to 100 °C	up to 50 bar	
>100 °C to 200 °C	up to 40 bar	
>200 °C to 250 °C	up to 30 bar	

This evaluation does not cover the use of the lubricant for liquid oxygen service. For this case, a particular test for reactivity with liquid oxygen needs to be carried out.

#### 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. 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, October 29, 2012

Division 2.1

"Gases, Gas Plante"

Dipl.-Ing. P. Hartwig

Study Director Safe Handling of Oxygen"

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- Working Group "Safe Handling of Oxygen" -

#### Annex 1

# <u>Determination of the Autogenous Ignition Temperature in High Pressure</u> <u>Oxygen</u>

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<sup>3</sup> 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 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<sup>3</sup> 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 tacAfter the tube and pipe are at test pressure pa, the quick opening valve is opened and preheated oxygen of 60 °C and of pressure pe 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 pa to pe 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<sub>e</sub>/p<sub>a</sub>. 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 pe/pa, 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 ta.