

Report

on Testing a Thread Sealant for Reactivity with Oxygen

Reference Number II-2287/2008 E
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1 Application

Customer Henkel AG & Co. KGaA
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TSI-Anwendungstechnik
Gutenbergstraße 3
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Order Date September 5, 2008

Reference Order-No.: 4591032091

Receipt of Order September 5, 2008

Test Samples Thread Sealant Loctite 55 for use in valves and fittings,
and other components for gaseous oxygen at tempera-
tures up to 60 °C, and with liquid oxygen.
BAM-Order No. II.1/49 406

Receipt of Samples June 18, 2008

Test Date October 23, 2008 to November 19, 2008

Test Location BAM - Working Group "Safe Handling of Oxygen";
building no. 41, room no. 120 and no. 073

**Test Procedure Ac-
cording to** DIN EN 1797: 2002-02
„Cryogenic Vessels - Gas/Material Compatibility“
Annex of pamphlet M 034-1 (BGI 617-1)
„Liste der nichtmetallischen Materialien die von der
Bundesanstalt für Materialforschung und -prüfung (BAM)
zum Einsatz in Anlageteilen für Sauerstoff als geeignet be-
funden worden sind.“,
to pamphlet M 034 „Sauerstoff“ (BGI 617)
Berufsgenossenschaft der chemischen Industrie
Edition: October 2007;
according chapter 3.17 „Gleitmittel und Dichtwerkstoffe“
to rule BGR 500 „Betreiben von Arbeitsmitteln“ part 2,
chapter 2.32 „Betreiben von Sauerstoffanlagen“,
Edition: April 2008.

All pressures in this report are excess pressures.
This test report consists of page 1 to 4 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.

TEST REPORT

2 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test Application
- 1 Safety Data Sheet
- 1 Material Data Sheet
- 2 packages Thread Sealant Loctite 55
 - à 150 m Thread sealing tape, diameter ca. 0,3 mm,
impregnated with silicone oil
Colour: white

3 Test Methods and Results

A determination of the autogenous ignition temperature (AIT) and a test for aging resistance in high pressure oxygen were not necessary as Loctite 55 is not for use at temperatures greater than 60 °C.

3.1 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1.

Results:

| Sample Temperature t_a [°C] | Oxygen Pressure p_a [bar] | Oxygen Pressure p_e [bar] | Reaction on Impact |
|----------------------------------|--------------------------------|--------------------------------|--------------------------|
| 60 | 1 | 30 | ignition on 2. impact |
| 60 | 1 | 25 | no reaction*) |
| 60 | 1 | 25 | ignition on 1. impact |
| 60 | 1 | 20 | no reaction*) |
| 60 | 1 | 20 | no reaction*) |

*) within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, with an initial pressure of $p_a = 1$ bar no reactions with oxygen could be observed for Loctite 55 at an oxygen pressure p_e of 20 bar.

3.2 Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 2.

Results:

| Test No. | Drop Heights [m] | Impact Energy [Nm] | Reaction |
|----------|---------------------|-----------------------|--------------------------------|
| 1 | 0,67 | 500 | violent reaction |
| 2 | 0,33 | 250 | violent reaction |
| 3 | 0,17 | 125 | violent reaction in 4. test |

At all drop heights of the plummet, reactions of Loctite 55 with liquid oxygen could be detected.

4 Evaluation

According to DIN EN 1797:2002-02 „Kryo-Behälter – Verträglichkeit von Gas/Werkstoffen“ the criterion for a positive reaction of the test material to gaseous oxygen impacts is a temperature rise of 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 thread sealant Loctite 55 in valves and fittings and other components for gaseous oxygen service at following operating conditions:

| | |
|------------------------------------|---|
| Maximum Temperature up to 60 °C | Maximum Oxygen Pressure up to 20 bar |
|------------------------------------|---|

According to the BAM-Standard “Testing for Reactivity with Liquid Oxygen on Mechanical Impact”, described in annex 2, the thread sealant material Loctite 55 is not suitable for liquid oxygen service.

5 Comments

The test results refer exclusively to the tested material.

Products that have been tested by us, and which are on the market, shall be marked according to our evaluation in the BAM test report. A label on a product saying that a BAM test has been performed and (or) citing our reference number, only, is not tolerable. The use of the product and its safe operating conditions must also be given.

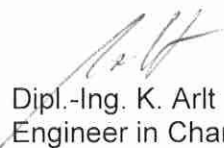
It shall be clear that the product may only be used for gaseous oxygen service. The maximum safe oxygen pressure of the product and its maximum use temperature as well as other restrictions in use shall be given.

BAM Federal Institute for Materials Research and Testing
12200 Berlin, February 19, 2009

Division II.1
"Gases, Gas Plants"


Dr. Chr. Binder
Head of Working Group

Working Group
"Safe Handling of Oxygen"


Dipl.-Ing. K. Arlt
Engineer in Charge

Copies: 1. Copy: Henkel KGaA
 2. Copy: BAM - Working Group "Safe Handling of Oxygen"

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 cm³ 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_a , the quick opening valve is opened and preheated oxygen of 60 °C and of pressure p_e 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_a to p_e 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_e/p_a . 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_e/p_a , 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 .

Annex 2

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.