Oxygen Compatibility Test Report

Materials:
DURLON® 9002 PTFE

Report Number:
R-WHA-16023-0-A-EX2

Date:
May 17, 2016

Prepared for:
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Project Details

WHA Project Number 16023-0

Client Triangle Fluid Controls Ltd.

Cost Estimate Number E-WHA-16023-0-A-EX1

Purchase Order Number BradM042216

Statement of Work WHA performed the following Oxygen Compatibility Testing on the listed materials, supplied by the client:

Material(s)	Test(s)
DURLON® 9002 PTFE	LOX Mechanical Impact Sensitivity

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Prepared by:	Reviewed by:	Approved by:
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DURLON® 9002

Material Details

WHA Test Article ID TA-16023-0-A1

Manufacturer Triangle Fluid Controls Ltd.

Material Type ☐Metal ☒Plastic ☐Elastomer ☐Grease ☐Liquid ☐Other

Trade Name DURLON® 9002

Generic Description 25% glass filled PTFE

Batch not available

Lot not available

Received Condition Material was received as preformed samples in the packaging as shown.

Tests Conducted ☒LOXMIS ☐GFIS ☐AIT ☐HoC ☐OI

LOX Mechanical Impact Sensitivity (LOXMIS)

Test Standard ASTM G86 - 98a (2011) - *Standard Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments*

Material Preparation Option A (**Table 3** in Appendix A)

Test Sample Details The average measurements for four randomly selected samples were:

- Mass: 0.422 g (0.0149 oz)
- Diameter: 0.660 in (16.8 mm)
- Thickness: 0.0380 in (0.965 mm)

Material Cleaning ☐Test as received ☒WHA (TPR-0030) ☐Other

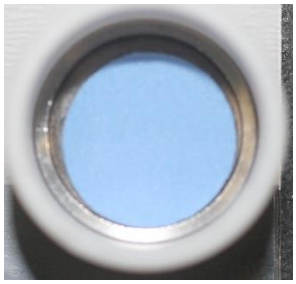
Test Conditions

- Test type: single level test
- Energy level: 125 J (92.2 ft·lbf) *

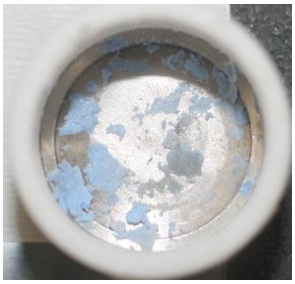
Test Results

- Passed with zero reactions in twenty tests (0 of 20)
- Test Reaction Frequency: 0 %
- Test Reaction Probability Range (see Appendix A §5): 0-17 %

Representative Photographs



(Pre-test)



(Post-test)

LOX Mechanical Impact Sensitivity (LOXMIS) *cont.*

Comments * ISO 21010 §4.4.3 requires mechanical impact testing in ambient pressure liquid oxygen to be conducted with a minimum energy level of 79 J/cm^2. This equates to an impact energy level of approximately 100 J using the test system specified by ASTM G86. ISO 21010 also specifies an acceptance criterion of no reactions within a series of ten tests (0 in 10). This is less severe that the ASTM G86 requirement of no reactions in twenty tests (0 in 20) or only one reaction in sixty tests (0 in 60).

Based on the test results, this test material has met or exceeded the pass/acceptance criteria for both ASTM G86 and ISO 21010.

Disclaimer

WHA International, Inc. does not endorse or warrant any component, item, or material tested by WHA personnel as being suitable for any design function or service application what-so-ever. WHA has not performed any evaluation or testing beyond that stated herein, and expressly denies any responsibility for having evaluated the test article or material for function or safety. WHA disavows any responsibility for the function or safety of test articles or materials.

Appendix A – WHA Test Description: Atmospheric Pressure Liquid Oxygen Mechanical Impact Sensitivity

1 Description

The atmospheric pressure Liquid Oxygen (LOX) Mechanical Impact Sensitivity (LOXMIS) test represents a test method to determine the relative sensitivity of materials to mechanical impact in liquid oxygen (LOX) and thereby provide an approach for ranking the compatibility of a material in LOX systems. WHA conducts LOXMIS material testing in accordance with either of the following standards: *1)* ASTM D2512[1], or *2)* ASTM G86[2]. LOXMIS testing is included in the preferred test methodologies indicated by ASTM G63[3].

1.1 Pass-Fail Testing

The Pass-Fail test method is described in ASTM D2512 §10.13 and ASTM G86 §5.3-6.3. Typically, testing for this method is conducted at the 98 J (72 ft·lbf) energy level. The result criteria specified in ASTM D2512 for this method require that at a selected energy level, a material exhibits no positive reaction in twenty tested samples (0 of 20). The result criteria specified in ASTM G86 requires that a material exhibit no more than one reaction in sixty tests (1 in 60) at the 98 J (72 ft·lbf) energy level and no reactions in twenty tests (0 in 20) for any energy level lower than 98 J.

1.2 Energy Screen Testing

The energy screen test method is described in ASTM D2512 §10.13-10.12 and ASTM G86 §13.5. For this test method a material is tested at multiple energy levels until the *energy threshold* is established. For ASTM G86, the highest energy level at which the required criteria are met is determined to be the *energy threshold*. For ASTM D2512, the higher of two sequential energy levels at which the passing criteria is met at both levels is determined to be the *definitive threshold value*.

1.3 Reaction Criteria

A positive reaction in LOXMIS testing is indicated by at least one of the following event/results: visible flash, audible report, evidence of burning (obvious charring, combustion odor[4], and/or significant discoloration).

2 Test System

The test system used by WHA is detailed in **Figure 1**. This system uses a 9.07 kg (20 lb) plummet mounted between to vertical guide rails. The energy level for each test is set by adjusting the initial drop height of the plummet to levels corresponding with **Table 1**.

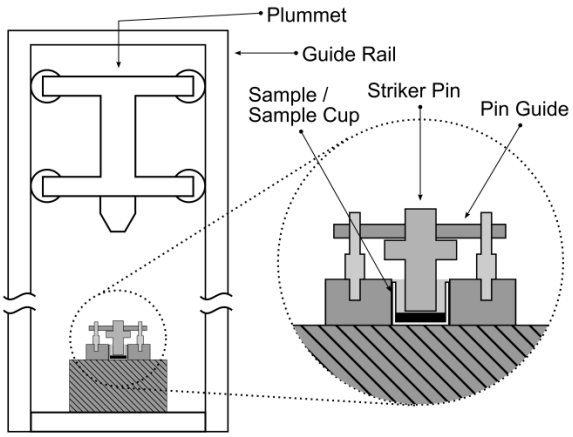


Figure 1 – WHA LOXMIS Test System Diagram

Table 1 – Drop Height Schedule for Energy Threshold Value Determination Using a 9.07-kg (20-lb) Plummet

Energy		Drop Height	
J	Ft·lbf	Meters	Inches
98	72	1.10	43.3
88	65	0.99	39.0
81	60	0.91	36.0
75	55	0.84	33.0
69	50	0.76	30.0
61	45	0.69	27.0
54	40	0.61	24.0
48	35	0.53	21.0
41	30	0.46	18.0
34	25	0.38	15.0
27	20	0.31	12.0
20	15	0.23	9.0
14	10	0.15	6.0

Typically, a two-piece sample cup (17-4 stainless steel base and PTFE sleeve) is used for holding the test samples.

2.1 System Calibration

The system performance for the LOXMIS test system is calibrated at regular intervals for each impact energy level used in testing by use of the dent block method described in both ASTM D2512 and ASTM G86. The dent

[1] ASTM Standard D2512, 95 (2008), "Standard Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)"
[2] ASTM Standard G86, 98a (2011), "Standard Test Method for Determining Ignition Sensitivity of Materials to Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments"
[3] ASTM Standard G63, 99 (2007), "Guide for Evaluating Nonmetallic Materials for Oxygen Service"
[4] Combustion odor alone is not sufficient to confirm a reaction.

Table 3 – Test Sample Preparation Options

Option	As-received Configuration	Fabrication Details
Solids		
A	Pre-fabricated test samples.	No fabrication required by WHA.
B	Any material configuration with dimensions sufficient for standard sample fabrication.	Standard samples are fabricated from the material. 17.5 mm (0.689 in) dia. disks (≤ 6.4 mm [0.25 in.] thick).
C	Any material configuration insufficient for standard sample fabrication.	The material is sectioned into small pieces 0.05-0.25 in. along each dimension and multiple pieces are combined to form individual test samples. The total mass of each test sample is 400-600 mg (0.0141 – 0.0212 oz).
Non-solids		
D	Material provided in the intended test condition or concentration.	Material is transferred directly to recessed sample cup (1.27 mm [0.050 in] depth, 0.362 mL [0.0122 fl. oz] approx. volume)
E	Material provided in unconcentrated condition and requires additional concentrating (for concentrated liquid sample testing or liquid residue testing)	Material is concentrated via rotary evaporation to the required concentration (2% of original volume). For residue, the concentrated material is further boiled off until only a residue remains.

block testing is used to ensure the system meets attenuation requirements. WHA's mechanical impact test system produces results consistent with NASA's published mechanical impact test system attenuation levels[5].

Test equipment used to measure critical test variables is calibrated, maintained, and used in testing according the WHA calibration and measurement procedures QPR-018, "Control of Monitoring and Measuring Equipment Procedure", and QPR-019, "Estimation of Uncertainty of Measurement Procedure". **Table 2** lists the critical test variables and the corresponding measuring equipment typically used in the mechanical impact test system.

Table 2 – Mechanical Impact Test Variable Measurement Equipment

Test Variable	Measuring Equipment	Specification	Uncertainty
plummet velocity	optical break-beam	0.01 to 10 m/s	± 0.05 m/s

3 Test Samples

The LOXMIS test system can accommodate most material types including: 1) metals, 2) plastics, 3) elastomers, 4) greases, 5) liquids, and 6) other materials. Test sample preparation and configuration is dependent on the type of material and the preliminary configuration of the material.

For solid materials, the ideal sample configuration is a 17.5 mm (0.689 in) diameter disc with a nominal thickness of 1.27 mm (0.050 in). Other thicknesses are allowed, however, the maximum allowable thickness is 6.4 mm (0.25 in). Additionally, if the material is used in parts or components with thin features less than 6.4 mm (0.25 in) thick, WHA recommends testing the material in its minimum use thickness. For test materials configured as a non-metallic coating on a metal substrate, the sample thickness should include the substrate at its minimum use thickness.

WHA further recommends that all test materials be cleaned for oxygen service before testing. If the material supplier cannot provide the test material/samples already cleaned, WHA can perform the cleaning before testing in accordance with the WHA cleaning procedure TPR-0030. However, WHA will also test materials in their as-received condition without cleaning if requested. Test samples, sample cups, and the tools used for sample preparation are always handled using clean handling procedures to avoid sample contamination.

Typically, WHA uses the standard preparation and configuration options shown above in **Table 3**.

4 Procedure

Prior to testing, the test samples are loaded into the standard two-piece sample cups (17-4 stainless steel base, PTFE sleeve) and the sample cups (along with striker pins) are placed in a chiller box cooled with LOX and allowed to completely freeze at atmospheric pressure LOX temperatures.

For each test, a sample is transferred from the chiller box and loaded into the LOXMIS test system. A pre-chilled striker pin is placed onto the sample and secured in position by the striker pin guide. LOX is then added

[5] J. W. Bransford, C. J. Bryan, G. W. Frye, and S. L. Stohler, "(NASA-TM-74106) LOX/GOX Mechanical Impact Tester Assessment," John F. Kennedy Space Center, NASA, Kennedy Space Center, FL, Technical Report 1980.

directly into the sample cup and sample cup holder until both are full. The test cell is then darkened and the plummet is dropped while the sample is still fully submerged in LOX.

Test operators monitor the test and observe any reactions that occur per the reaction criteria in previously in **§1.3** of this test description. Test parameters, observations, and results are recorded for each test.

5 Results and Analysis

For each tested material, one or multiple of the following results are reported:

1) *Pass-Fail Status* – The resulting pass/fail status of a test material at the tested energy level with respect to the criteria set forth in ASTM D2512 (see previous section **§1.1**).
2) *Energy Threshold* – The highest energy level at which a test material meets the required criteria set forth in ASTM D2512 or ASTM G86 (see previous section **§1.2**).
3) *Test Reaction Frequency* (%) – The number of observed reaction results divided by total number of tests conducted and multiplied by 100 (calculated per energy level).
4) *Logistic Regression Chart* – The resulting chart from a logistic regression analysis of the binomial test data with 95% confidence intervals.
5) *Test Reaction Probability Range* (% - %) – The 95% confidence interval calculated from the number of observed reaction results and the total number of tests using the Clopper-Pearson ExactCI method[6].

When sufficient testing is performed to obtain an impact energy screen or pressure screen, a logistic regression analysis of the test data is conducted and the resulting logistic regression chart is included in the material test report. The logistic regression analysis is performed according to ASTM G74 §10.1.3[7]. When available, a logistic regression chart for a reference (previously tested) material is included for comparison. If testing produces insufficient test data to complete a logistic regression analysis, or if testing was only conducted at a single energy level (such as in Pass-Fail testing), the Test Reaction Probability Range for each tested energy level is reported instead of the logistic regression chart.

[6] C. Clopper and E. S. Pearson, *The Use of Confidence or Fiducial Limits Illustrated in the Case of the Binomial*.: Biometrika 26, 1934, pp. 404-413.

[7] ASTM G94 – 13, "Standard Test Method for Ignition Sensitivity of Nonmetallic Materials and Components by Gaseous Fluid Impact"