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Determination of Autogenous Ignition Temperature for Isopropyl Alcohol and Ethanol

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July 2020

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The assessment team would like to acknowledge the significant contributions made by John Bouvet, Jose Luis Alday, and George Quezada in the generation of the data.

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NASA Engineering and Safety Center
Technical Assessment Report

Determination of Autogenous Ignition Temperature for Isopropyl Alcohol and Ethanol

June 18, 2020

Report Approval and Revision History

NOTE: This document was approved at the June 18, 2020, NRB. This document was submitted to the NESC Director on June 22, 2020, for configuration control.

Approved:	*Original Signature on File*	6/24/20
	NESC Director	Date

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1.0	Initial Release	Dr. Daniel J. Dorney, NASA Technical Fellow for Propulsion, Marshall Space Flight Center (MSFC)	6/18/20

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Technical Assessment Report

1.0 Notification and Authorization

As part of a recent liquid rocket engine shutdown investigation, a commercial partner requested that NASA provide the autogenous ignition (or autoignition) temperature (AIT) of isopropyl alcohol (IPA). NASA provided the available data, but the data were somewhat scattered, likely due to test configuration and test technique differences. NASA Engineering and Safety Center (NESC) support was requested to experimentally determine the AIT of IPA and ethanol, both of which are extensively applied to propulsion systems.

Key stakeholders for this assessment included the Commercial Crew Program (CCP) and other programs using isopropyl alcohol and ethanol.

2.0 Signature Page

Submitted by:

Team Signature Page on File – 7/2/20

Dr. Daniel J. Dorney Date

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Ms. Susana A. Harper Date	Mr. Mark B. McClure Date
Ms. Ilse A. Reyes Date	Mr. Alfredo Juarez Date
Dr. Daniel J. Wentzel Date	

Signatories declare the findings, observations, and NESC recommendations compiled in the report are factually based from data extracted from program/project documents, contractor reports, and open literature, and/or generated from independently conducted tests, analyses, and inspections.

3.0 Team List

Name	Discipline	Organization
Core Team		
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Betty Trebaol	Project Coordinator	LaRC/AMA

3.1 Acknowledgments

The assessment team would like to acknowledge the significant contributions made by John Bouvet, Jose Luis Alday, and George Quezada in the generation of the data.

4.0 Executive Summary

As part of a recent liquid rocket engine shutdown investigation, a commercial partner requested that NASA provide the autoignition temperature (AIT) of isopropyl alcohol (IPA) in a pure oxygen environment. Although NASA provided the available data, the data showed variability between data sources. The available data were for much lower pressures than required, and the majority of the data were for air rather than oxygen. The scatter seen in previous tests was likely due to test configuration and experimental technique differences, as well as inherent variability in the AIT response itself. NASA Engineering and Safety Center (NESC) support was requested to experimentally determine the AIT of both IPA and ethanol, both of which are extensively applied to clean and flush propulsion systems.

Autoignition testing of IPA and ethanol was performed at White Sands Test Facility (WSTF) for pressures representative of those found in spacecraft and launch vehicle propulsion systems. Five tests were performed at each pressure, and the results are summarized below:

- The average AITs for IPA in gaseous oxygen at 10.3 megapascals (MPa) (1,500 pounds force per square inch (psi)) and 15.2 MPa (2,200 psi) were 199.3 degrees Celsius (°C) (390.8 degrees Fahrenheit (°F)) and 201.6 °C (394.8 °F), respectively.
- The average AITs for ethanol in gaseous oxygen at 10.3 MPa (1,500 psi) and 15.2 MPa (2,200 psi) were 193.2 °C (379.8 °F) and 198.2 °C (388.8 °F), respectively.
- The delta pressure rise associated with the autoignition of IPA increases nonlinearly as the mass of the IPA is increased.
- The delta pressure rise associated with autoignition is greater in IPA than in ethanol.

5.0 Assessment Plan

The scope of this assessment was to determine the AIT of liquid IPA and liquid ethanol in gaseous oxygen. The tests were performed at 10.3 and 15.2 (MPa) (1,500 and 2,200 psi). The pressures were chosen to enable comparisons with previous tests and to provide data at relevant propulsion system operating conditions. The tabulated data will be provided to the CCP and other interested programs/projects across NASA.

6.0 Solution Description

IPA and ethanol AITs were determined for two pressures (i.e., 10.3 MPa and 15.2 MPa/ 1,500 and 2,200 psi). Each test condition was repeated five times with 200 milligrams (mg) of IPA and ethanol, and the AITs were averaged. One test case was performed at 10.3 MPa (1,500 psi) using 500 mg of IPA.

7.0 Data Analysis

Limited data are available in the literature for the AITs of IPA and ethanol. Table 7.0-1 shows some of the properties for IPA and ethanol in nominal 21% oxygen air at approximately 14.7 pounds force per square inch absolute (psia) (i.e., sea level) and room temperature [ref. 2].

Table 7.0-1. IPA and Ethanol Properties from CRC[1] Tables

	IPA	Ethanol
Boiling point (°C/°F)	82.3/180.1	78.2/172.8
Flammability limit (%)	2.0–12.7	3.3–19.0
AIT (°C/°F in air)	399/750.2	363/685.4
Flash point (°C/°F)	12/53.6	13/55.4

Table 7.0-2 contains additional data for ethanol in normal pressure (14.7 psia) air and oxygen [ref. 3]. The differences in the AITs for ethanol in Tables 7.0.1 and 7.0.2 may be caused by test configuration and technique differences or by the quality of the ethanol used. The variations may also be caused by variability of the tests in general. These tables underscore the need for systematic testing to generate AIT data sets for IPA and ethanol. No data at higher pressures for either solvent were found during the NESC assessment team's literature search.

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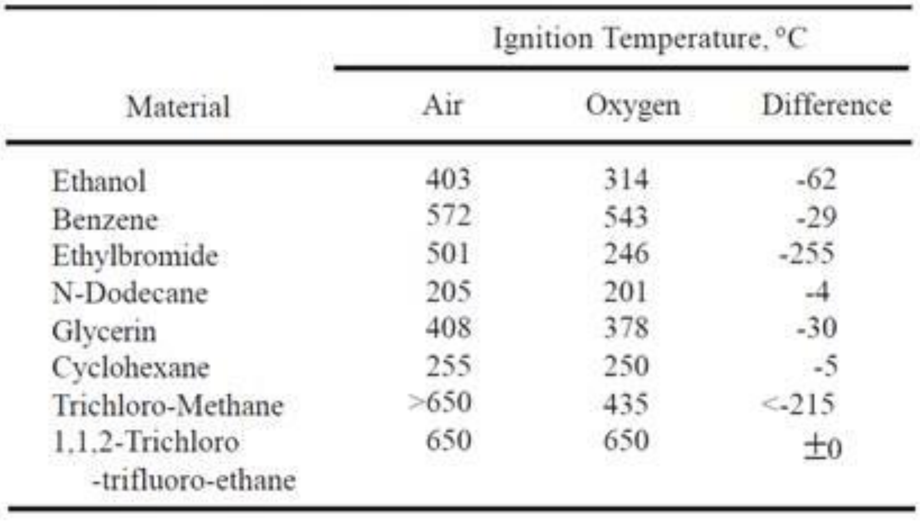
[1] CRC originally stood for Chemical Rubber Company, but in 1973, the company changed its name to CRC Press, Inc.

Table 7.0.2. Normal Pressure Data for Ethanol



Material	Ignition Temperature, °C		
	Air	Oxygen	Difference
Ethanol	403	314	-62
Benzene	572	543	-29
Ethylbromide	501	246	-255
N-Dodecane	205	201	-4
Glycerin	408	378	-30
Cyclohexane	255	250	-5
Trichloro-Methane	>650	435	<-215
1,1,2-Trichloro -trifluoro-ethane	650	650	±0

8.0 AIT Testing

AIT tests are performed as a means of determining the temperature at which liquids and solids spontaneously ignite. The standard is detailed in American Society for Testing and Materials (ASTM) G72. For this assessment, the AIT testing was performed at WSTF. Before 2015, the ASTM G72 test method did not provide guidance on how to best approach the testing of volatile materials. Volatile materials evaporate quickly during test setup and testing, presenting a challenge to the test lab. Labs varied in their approaches on how to best perform AIT testing on volatile materials. Some labs took extensive measures to minimize evaporation and increased initial sample sizes to account for expected losses, while others tested per typical standard material protocols. Differences in handling and testing approaches are likely to have contributed to variations in community test data.

In 2015, the NASA WSTF Flammability Test Team worked with ASTM to incorporate direction on how to best approach and test volatile materials to ensure the generation of valid and consistent data. The current G72-15 includes this guidance. Beyond the recommended volatile material guidelines set forth in the G72-15 document, WSTF takes additional precautions to minimize material loss and ensure valid data. The testing performed for this assessment was conducted in accordance with the volatile material guidelines outlined in G72-15, as well as with WSTF’s additional precautions for testing of volatile materials.

8.1 Preparation Method – AIT Testing

Volatile materials evaporate quickly during test setup and testing, presenting a challenge to the test lab. To limit the evaporation of test material, special handling was required. Glass beakers of the solvent were chilled in a refrigerator. Once a material was weighed, the preweighed amount of the chilled solvent was maintained by nesting the test vial within a 20-milliliter (mL) glass vial partially submerged in a saltwater ice bath.

8.2 Test Method – AIT Testing

The WSTF standard test method is performed as follows. A sample holding assembly, contained within a reaction vessel pressurized with 100% oxygen to 10.3 MPa (1,500 psi), is heated in an electric furnace at a rate of 5 ± 1 °C (9 ± 1 °F)/min from 60 to 260 °C (140 to 500 °F). Heating of the vessel is continued at an uncontrolled rate to a maximum temperature of 450 °C (842 °F). Temperatures are monitored as a function of time by means of a thermocouple and data acquisition system. During testing, pressure is monitored but not maintained. Ignition of the test sample is indicated by a rapid temperature rise of at least 20 °C (36 °F) and is confirmed post-test by the destruction of the sample.

Standard testing is performed at 10.3 MPa (1,500 psia) with a sample size of 200 ± 30 mg. A standard sample size of 200 ± 30 mg is selected to prevent damage to the test apparatus as a result of an overpressure caused by a reaction of the test material. Nonetheless, larger sample sizes are sometimes required when testing volatile materials to achieve a reaction. Samples are typically maintained at room temperature, but volatile materials are chilled as described in Section 8.1. Three purges of 100% oxygen at a minimum of 345 kilopascals (kPa) (50 psia) are performed prior to each test to ensure the venting of residual air.

Deviation from ASTM G72 pressures was requested of WSTF to coincide with anticipated system operating conditions. AIT tests were also performed at 15.2 MPa (2,200 psia).

8.2.1 Test Sample Size

Any combustion event requires three components: fuel, oxidizer, and ignition source. Autogenous ignition temperature tests are typically performed in 100% oxygen environments (oxidizer), and the test method provides the heat/ignition source; therefore, ensuring adequate combustible material is key to combustion.

Volatile solvents present a challenge prior to testing; however, once the reaction chamber is sealed, the volatility should not present a problem in igniting the material. These materials all burn in the gas phase; therefore, evaporation of the material is required for combustion. Standard size samples may be an insufficient amount of material to react violently enough to register as an AIT.

Addressing inadequate sample size normally involves gradually increasing the sample size until a reaction is observed. This gradual increase in sample size allows for a controlled method of varying the test protocol while ensuring safe operation of the test system. In the current tests, 200 mg was determined to be sufficient for the testing, although one test was performed using 500 mg of IPA for comparison purposes.

8.2.2 Pretest Purges

The potential for forced evaporation due to pretest purging was considered and evaluated for both solvents. Two types of tests were performed to evaluate the effect of purging on the AIT test results.

The first test used to evaluate purging consisted of pressurizing test samples immediately followed by removal of the test samples. No heat was induced on samples during these simulation tests. Samples were chilled and weighed prior to loading into a reaction vessel. Three consecutive pressurization/vent cycles were completed prior to removal of the test sample. Posttest weights were recorded and compared with pretest weights.

The second evaluation conducted to investigate this variable involved performing AIT tests with and without purges. For this test series, purges were determined to be acceptable. Ethanol and IPA AIT tests were performed at each of the requested pressures using three test pressure purges.

8.2.3 Thermocouple Placement

The position of the thermocouple was examined to determine its influence on AIT results. Figure 8.2.3-1 is a schematic of the AIT assembly. The standard depth of the tip of the thermocouple relative to the sample holder is 15 millimeters (mm) below the upper edge of the test vial. The “X” dimension shown in Figure 8.2.3-1 represents the length that was varied. Tests were performed to examine this variable at the standard depths of 15, 30, and 60 mm (0.59, 1.18, and 2.36 inches). All tests were performed using three purges and 200-mg samples. The thermocouple position was not found to have an influence on AIT results. All testing presented in this paper was performed with the thermocouple at the standard depth of 15 mm.

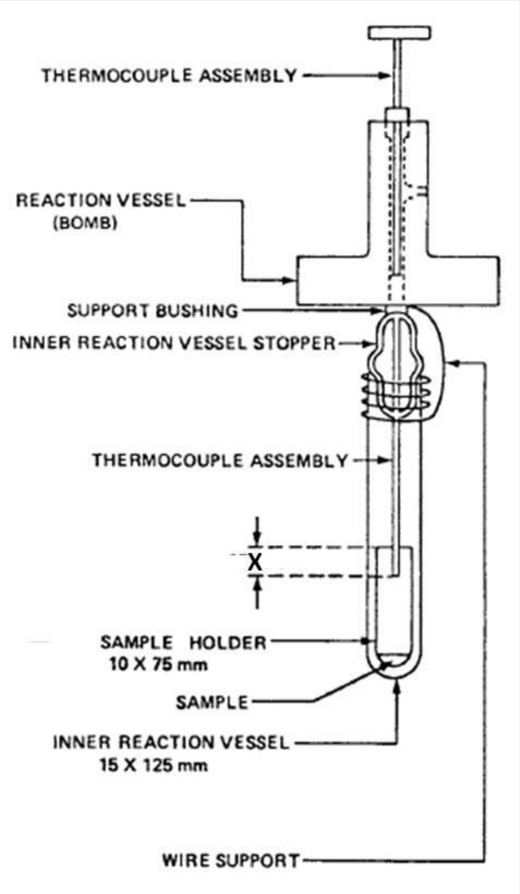


Figure 8.2.3-1. AIT Test Assembly

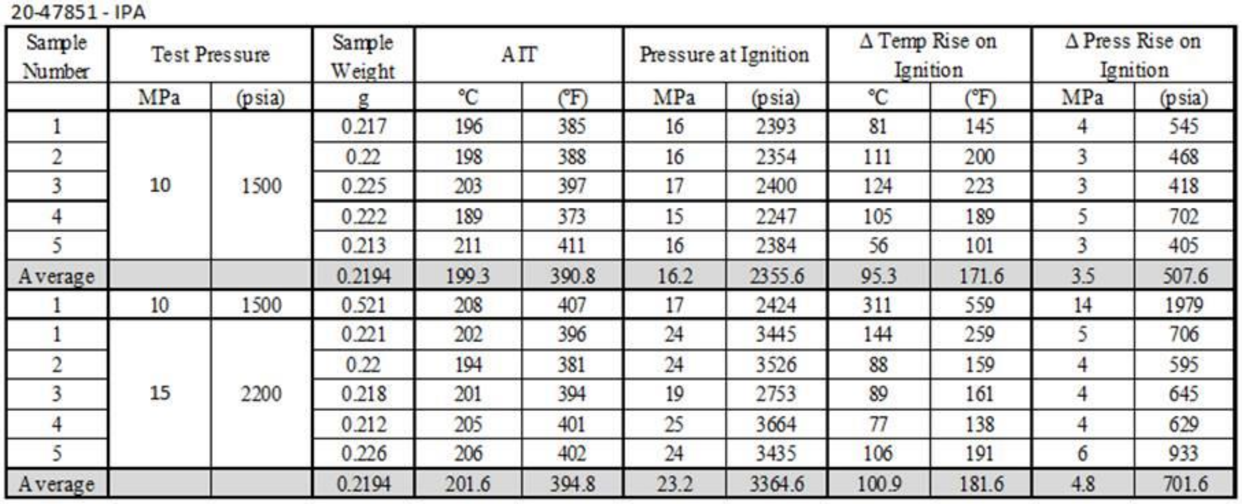
8.3 IPA and Ethanol Tests

The tests used Sigma-Aldrich anhydrous 2-propanol (IPA), part number 278475, 99.5% purity, and Sigma-Aldrich ethyl alcohol (ethanol), pure, part number 459844, minimum 99.5% purity, American Chemical Society (ACS) reagent. Both the IPA and ethanol were used as received without further purification. Testing was performed for the IPA and the ethanol at both 10.3 MPa (1,500 psi) and 15.2 MPa (2,200 psi). Five tests were run at each pressure using ~200 mg each of the IPA and ethanol. An additional test was run using 500 mg of IPA at 1,500 psi.

Tables 8.3-1 and 8.3-2 show a summary of the test data for the AITs and pressures for the IPA and ethanol. The average AITs for IPA at 10.3 MP (1,500 psi) and 15.2 MPa (2,200 psi) were 199.3 °C (390.8 °F) and 201.6 °C (394.8 °F), respectively. The AIT for the 1,500 psi test using 500 mg of IPA was encompassed by the AIT values obtained using 200 mg. Note the delta pressure rise associated with the autoignition of the IPA (last column of Table 8.4-1) for the 500-mg test case, which is nearly four times greater than the average of the 200-mg cases.

The average AITs for ethanol at 10 MPa (1,500 psi) and 15 MPa (2,200 psi) were 193.2 °C (379.8 °F) and 198.2 °C (388.8 °F), respectively. The AITs for ethanol are close to those for IPA, albeit slightly lower. The pressures at ignition are also similar to those for IPA. The delta pressure rise at ignition, however, is greater for IPA than for ethanol. This makes sense, as 200 mg of IPA should create about 6.6 kJ of heat compared with ethanol’s 6.0 kJ of heat.

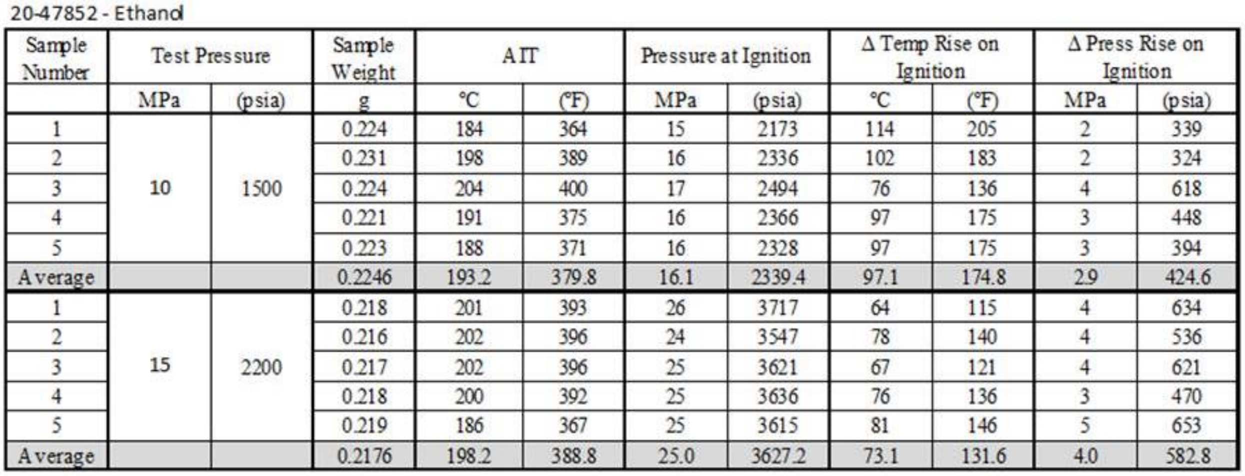
Table 8.3-1. Autoignition Temperature and Pressure Data for IPA



20-47851 - IPA

Sample Number	Test Pressure		Sample Weight	AIT		Pressure at Ignition		Δ Temp Rise on Ignition		Δ Press Rise on Ignition	
	MPa	(psia)	g	°C	(°F)	MPa	(psia)	°C	(°F)	MPa	(psia)
1	10	1500	0.217	196	385	16	2393	81	145	4	545
2			0.22	198	388	16	2354	111	200	3	468
3			0.225	203	397	17	2400	124	223	3	418
4			0.222	189	373	15	2247	105	189	5	702
5			0.213	211	411	16	2384	56	101	3	405
Average			0.2194	199.3	390.8	16.2	2355.6	95.3	171.6	3.5	507.6
1	10	1500	0.521	208	407	17	2424	311	559	14	1979
1	15	2200	0.221	202	396	24	3445	144	259	5	706
2			0.22	194	381	24	3526	88	159	4	595
3			0.218	201	394	19	2753	89	161	4	645
4			0.212	205	401	25	3664	77	138	4	629
5			0.226	206	402	24	3435	106	191	6	933
Average			0.2194	201.6	394.8	23.2	3364.6	100.9	181.6	4.8	701.6

Table 8.3-2. Autoignition Temperature and Pressure Data for Ethanol



20-47852 - Ethanol

Sample Number	Test Pressure		Sample Weight	AIT		Pressure at Ignition		Δ Temp Rise on Ignition		Δ Press Rise on Ignition	
	MPa	(psia)	g	°C	(°F)	MPa	(psia)	°C	(°F)	MPa	(psia)
1	10	1500	0.224	184	364	15	2173	114	205	2	339
2			0.231	198	389	16	2336	102	183	2	324
3			0.224	204	400	17	2494	76	136	4	618
4			0.221	191	375	16	2366	97	175	3	448
5			0.223	188	371	16	2328	97	175	3	394
Average			0.2246	193.2	379.8	16.1	2339.4	97.1	174.8	2.9	424.6
1	15	2200	0.218	201	393	26	3717	64	115	4	634
2			0.216	202	396	24	3547	78	140	4	536
3			0.217	202	396	25	3621	67	121	4	621
4			0.218	200	392	25	3636	76	136	3	470
5			0.219	186	367	25	3615	81	146	5	653
Average			0.2176	198.2	388.8	25.0	3627.2	73.1	131.6	4.0	582.8

The ASTM G72 apparatus is stated to have a volume of 110 mL. Assuming that some volume is lost to internal hardware, a 100-mL (0.1 liter) volume can be used for calculation purposes. The starting temperature was assumed to be 20 °C, with pressures at 1,500 and 2,200 psia for an ideal gas law calculation, which produced 0.424 and 0.623 moles (mol) of oxygen, respectively, for those pressures with the amount of oxygen available. A 0.200-g ethanol sample at 46 g/mol for 0.0044 mol and 0.200 grams of IPA at 60 g/mol for 0.0033 mol of IPA were assumed.

Reactions balanced for complete combustion (no CO, all CO_2):

Ethanol: $C_2H_6O + 3O_2 \rightarrow 2CO_2 + 3H_2O$

IPA: $C_3H_8O + 4.5O_2 \rightarrow 3CO_2 + 4H_2O$

These reactions and molar quantities show that this is an extreme oxidizing environment with an oxygen molar excess (0.424 mol of oxygen available with 0.0132 and 0.0148 mol consumed for ethanol and IPA, respectively). For the ethanol example at 1,500 psia, this also means the molar increase in volume of $0.424 + 0.0044 \rightarrow 0.424 - 0.0132 + 0.0088 + 0.0132$ for a net 0.428 to 0.433 mol total is not making the pressure of note based on volumetric expansion alone.

As this is a combustion reaction, the heat of combustion for ethanol (continuing the example above) was 1,370 kJ/mol and, with 0.0044 mol available, generated 6.028 kJ of heat. Assuming adiabatic conditions and mostly oxygen (as noted in the oxygen-to-fuel ratio):

- The heat capacity for oxygen is 0.659 kJ/kg-K (or 0.659 J/g-K).
- The (0.433 mol total) × (32 g/mol) results in 13.85 g of oxygen.
- Finally, (6,028 J / 0.659 J/g-K) × 13.85 g oxygen = 660 K as the ΔT.

This change in temperature would not be expected, but demonstrates that the pressure rise is due to combustion heating of the gas in the vessel. Another indication that this assumption is not completely sound is that extra diluent oxygen would lower the temperature and the pressure rise data, but this is not observed at all in the pressure data and only in the temperature data for ethanol. The assumption that complete combustion occurs might be erroneous as well. Measuring transient temperatures with a thermocouple is notoriously inaccurate, so the ~100 °C rise instead is reasonable. The fact that 500 mg is a factor 2.5 times the 200 mg and the heat is increased only about 2.5 times may cause concern; how that heat translates into P and T in the test system with heat transfer, combustion efficiency, and other unknowns is of interest. In the end, recognizing that to be the case, the test team used only the finding of a temperature and pressure rise as an indicator of combustion initiation and not the final readings. Final temperature and pressure values are of most value in protecting the system from disassembly as the tests are run.

8.4 Statistical Analysis of AIT, Pressure, and Temperature Data

To further investigate the effects of fluid, test pressure, and sample mass, a statistical analysis was carried out using JMP statistical analysis software [ref. 4]. Fluid, test pressure, and sample mass were identified as independent variables, or factors (i.e., explanatory variables that can be changed by the experimenter). Fluid was a categorical factor of the two types (i.e., IPA and ethanol). Test pressure was a continuous numeric factor of two levels (1,500 and 2,200 psia). Sample mass was another continuous numeric factor; this was not varied to a large degree, except for one IPA test at approximately twice the mass. Four dependent responses (i.e., variables that respond to changes in the factors) were also monitored during the testing: AIT, pressure at ignition, change in temperature, and change in pressure. Pressure at ignition was not analyzed because it was found to be highly correlated with test pressure.

8.4.1 Autoignition

Determining the AIT of ethanol and IPA was the primary goal of this assessment. A large amount of variability existed in the AIT data. This made analysis challenging because it was difficult to separate the signal from the noise. Based on the data collected, the only significant factor was the fluid. The comparison of the AITs of ethanol and IPA are shown in graphically in Figure 8.4.1-1, and a comparison of the quantiles is shown in Table 8.4.1-1. The solid horizontal line in Figure 8.4.1-1 (at slightly less than 390 °F) represents the mean of the data.

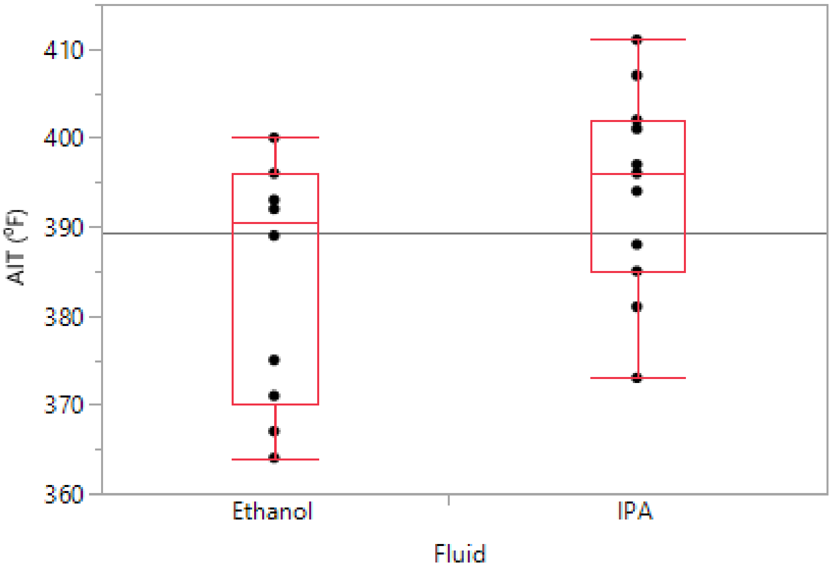
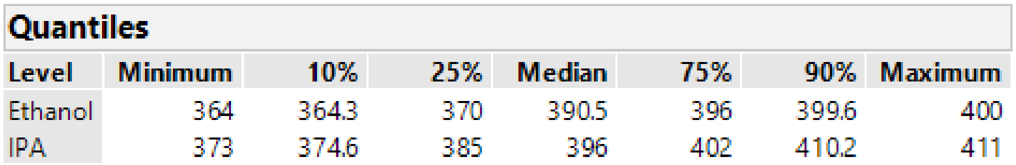


Figure 8.4.1-1. Box and Whiskers Display of AIT Data

Table 8.4.1-1. Quantile Temperature (°F) Values for Data Collected

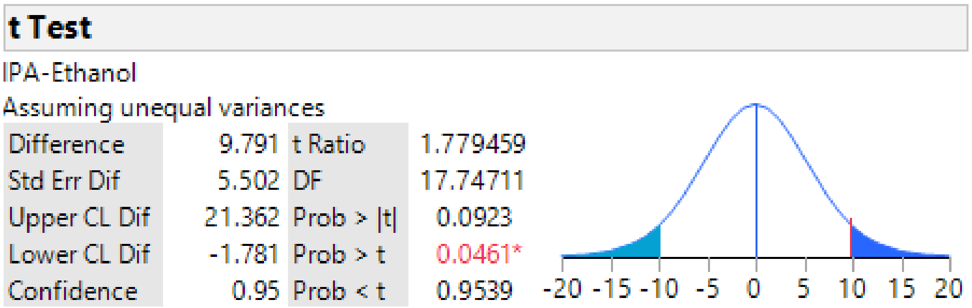


Quantiles

Level	Minimum	10%	25%	Median	75%	90%	Maximum
Ethanol	364	364.3	370	390.5	396	399.6	400
IPA	373	374.6	385	396	402	410.2	411

As can be seen in the plot, the data show significant overlap, and the difference between the two data sets is very small. To determine whether this difference is real, a statistical test was performed to compare the two means. A t-test was used to do this comparison, and the results are shown in Table 8.4.1-2.

Table 8.4.1-2. Output Results of Performed t-test



```
t Test
IPA-Ethanol
Assuming unequal variances
Difference       9.791  t Ratio     1.779459
Std Err Dif      5.502  DF          17.74711
Upper CL Dif    21.362  Prob > |t|    0.0923
Lower CL Dif    -1.781  Prob > t      0.0461*
Confidence        0.95  Prob < t      0.9539
```

The conclusive feature in the t-test is the value “Prob > t.” The literature indicates that the AIT for IPA is greater than that of ethanol. Therefore, it can be reasonably concluded that a single-sided t-test is applicable. The test indicates, based on the data collected, that IPA does have a statistically significant higher AIT than ethanol.

8.4.2 Pressure Change

Another important goal of this study was to observe the pressure change because of the autoignition event. A statistical model of the change in pressure was developed. As mentioned previously, the mass of the sample used was identified as a significant driver of pressure because of the 500-mg test point. This data point proved troublesome in the analysis because it is a high leverage point—meaning it is far away from the majority of the data and accounts for only one combination of factors. Therefore, the variability is artificially low and can have a significant negative effect on the ability to develop a realistic model. Fortunately for this response, the inclusion of this data point added information and did not negatively affect the model. Based on the data collected, the three main factors were all significant (i.e., sample weight, test pressure, and fluid). The actual values are compared with the estimated statistical model predictions in Figure 8.4.2-1.

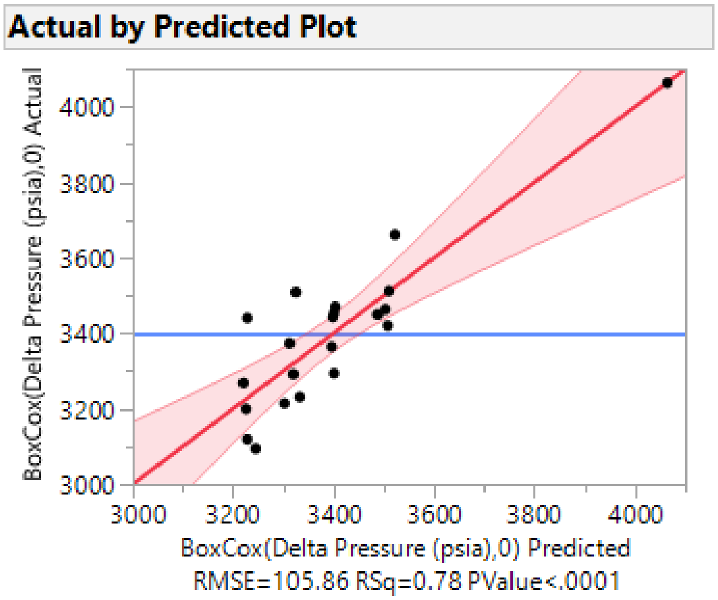


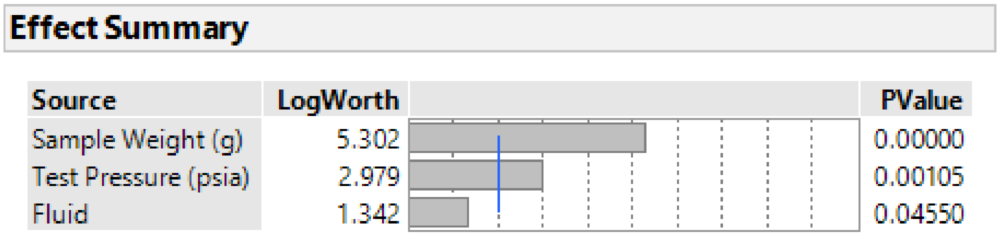
Figure 8.4.2-1. Actual Values versus Model Predictions for Test Points

A log transform was used on the data to create a model that better fit the data. Similar to how a coordinate transformation is used in mathematics to work in a natural frame of reference, statistical transformations can be used in cases where data trends are nonlinear (e.g., log, exponential, inverse, etc.).

Figure 8.4.2-1 shows the R-squared for this model is 0.78. This means that the statistical model can explain 78% of the total variability of the data. The closer the value is to 1, the higher the explanatory power of the model. A value of 0.78 is not completely informative because the model cannot explain 22% of the data variation; however, given how noisy the response values were, it is determined acceptable to identify the important trends and significant factors.

The effects summary and the parameter estimate table gives values for the associated significant model terms (see Tables 8.4.2-1 and 8.4.2-2). The p-value is used to determine whether an effect is statistically significant. Note that in statistics the p-value or probability value is the probability of obtaining test results at least as extreme as the results actually observed during the test. For this analysis, a significance level of 0.05 was used (for a term to be included in the model, the p-value had to be less than 0.05). The parameter estimate table gives the values of the coefficients making up the linear model that describes the data.

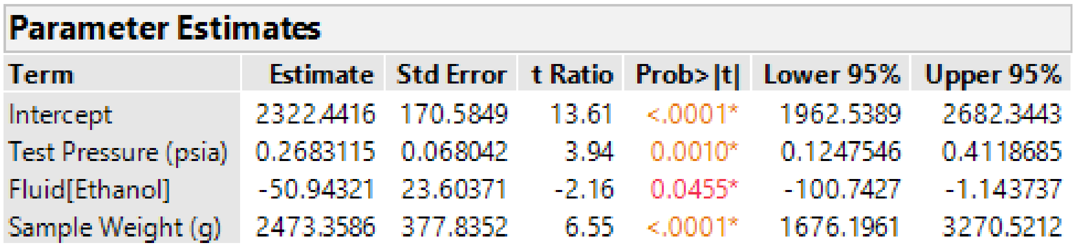
Table 8.4.2-1. List of Factors in Model with Significance Values



Effect Summary

Source	LogWorth	PValue
Sample Weight (g)	5.302	0.00000
Test Pressure (psia)	2.979	0.00105
Fluid	1.342	0.04550

Table 8.4.2-2. Model Parameter Estimates



Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|	Lower 95%	Upper 95%
Intercept	2322.4416	170.5849	13.61	<.0001*	1962.5389	2682.3443
Test Pressure (psia)	0.2683115	0.068042	3.94	0.0010*	0.1247546	0.4118685
Fluid[Ethanol]	-50.94321	23.60371	-2.16	0.0455*	-100.7427	-1.143737
Sample Weight (g)	2473.3586	377.8352	6.55	<.0001*	1676.1961	3270.5212

The prediction profiler describes, within the design space, where the data should lie as a function of the modeled terms (see Figure 8.4.2-2). There is an increasing trend in the change in pressure as a function of both test pressure and sample weight. This means that as either the test pressure or the sample weight is increased or decreased, the change in pressure will increase or decrease accordingly. Additionally, the change in pressure is a function of the fluid. By comparison, ethanol has a smaller change in pressure than IPA.

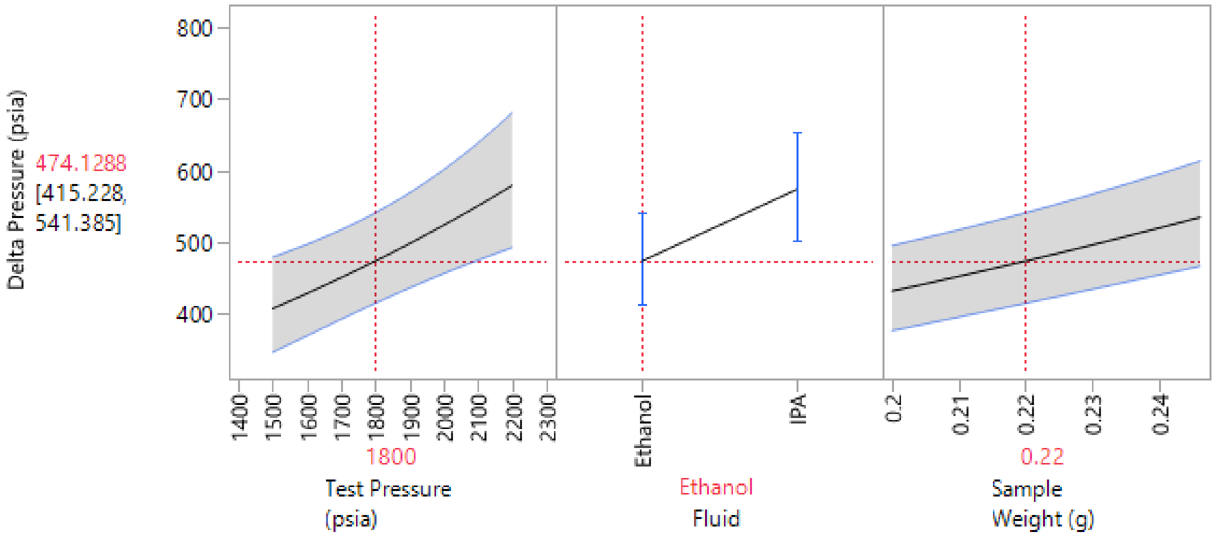


Figure 8.4.2-2. Prediction Profiler of Statistical Model

A note about prediction: while the 500-mg data point was used to develop the model, it would be inappropriate to use this model to predict above where the majority of the data were taken (around 220 mg). Because there was only one data point at 500 mg, it may be an extrapolation to try to predict in that space.

This model suggests that, to minimize the change in pressure, systems should be operated at lower pressures with smaller amounts of fluid. Further, if given the option between ethanol and IPA, ethanol is a better choice in terms of the change in pressure.

8.4.3 Temperature Change

A final goal of this study was to observe the temperature change due to the autoignition event. A statistical model of the change in temperature was developed. The high leverage point (500 mg IPA) was removed for this analysis. As mentioned earlier, this high mass point had the potential to have a significant negative effect on the development of a realistic model. This was found to be the case for change in temperature. The point was different enough from the majority of the data points that it drove the model to explain the outlier and not the majority. For that reason, this data point was excluded. Based on the data collected, the primary effects of sample weight and fluid were all significant. The actual values are compared with the estimated statistical model predictions in Figure 8.4.3-1.

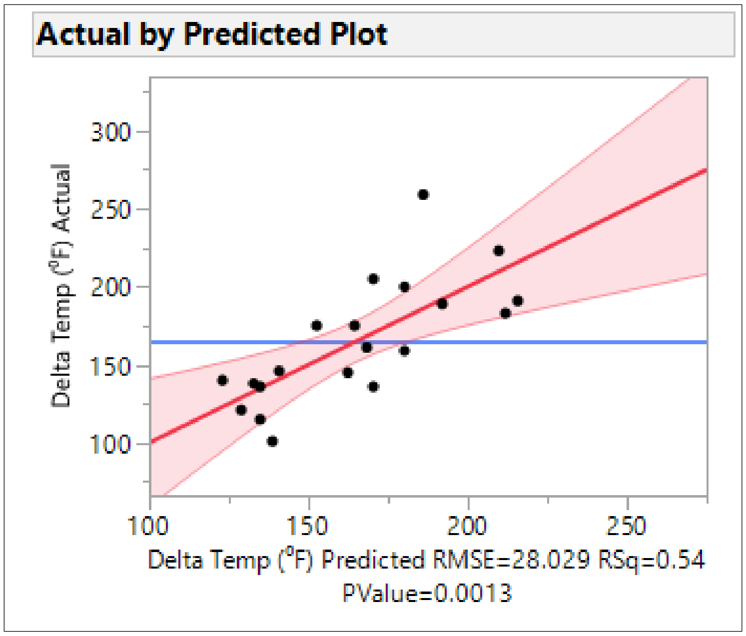
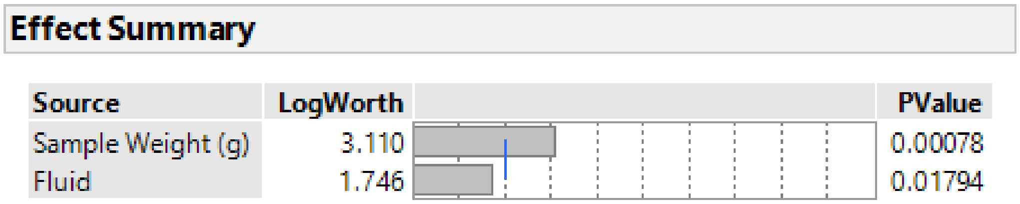


Figure 8.4.3-1. Actual Values versus Model Predictions for Test Points

Observe from Figure 8.4.3-1 that the R-squared for this model is 0.54, meaning that the model can explain 54% of the total variability of the data. In general, R-squared is a statistical measure of how close the data are to the fitted regression line. A value of 0.54 indicates that while the model has significant explanatory capability over the range of inputs (see Table 8.4.3-1), unexplained variation in change in temperature is quite large compared with the effects of those inputs on the response. As previously mentioned, the data are quite noisy, and that noise shows up in the 46% that the model cannot explain. However, the model is sufficient to identify the important trends and significant factors out of the list of those tested.

The effects summary and the parameter estimate tables (Tables 8.4.3-1 and 8.4.3-2) give values for the associated significant model terms. The p-values for the terms in the statistical model were all at a significance level of 0.05, and the parameter estimate table gives the values of the coefficients that make up the linear model describing the data.

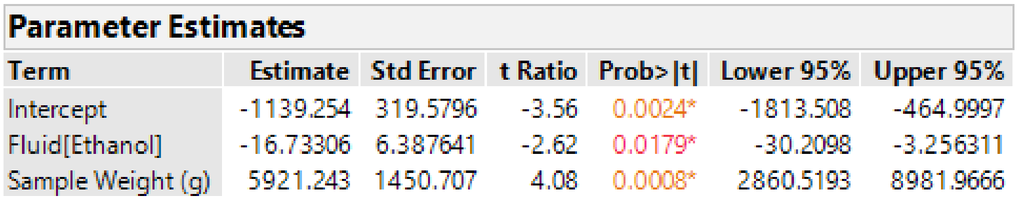
Table 8.4.3-1. List of Factors in Model with Significance Values



Effect Summary

Source	LogWorth		PValue
Sample Weight (g)	3.110		0.00078
Fluid	1.746		0.01794

Table 8.4.3-2. Model Parameter Estimates



Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|	Lower 95%	Upper 95%
Intercept	-1139.254	319.5796	-3.56	0.0024*	-1813.508	-464.9997
Fluid[Ethanol]	-16.73306	6.387641	-2.62	0.0179*	-30.2098	-3.256311
Sample Weight (g)	5921.243	1450.707	4.08	0.0008*	2860.5193	8981.9666

The prediction profiler describes within the design space where the data should lie as a function of the modeled terms. There is an increasing trend in the change in temperature as a function of sample weight (meaning that as the sample weight is increased or decreased, the change in temperature will increase or decrease accordingly). Additionally, the change in temperature is also a function of the fluid type. By comparison, ethanol has a smaller change in temperature than IPA.

This model suggests that, to minimize change in temperature, systems should be designed with smaller amounts of fluid (see Figure 8.4.3-2). Further, if given the choice between ethanol and IPA, ethanol is a better option in terms of temperature change.

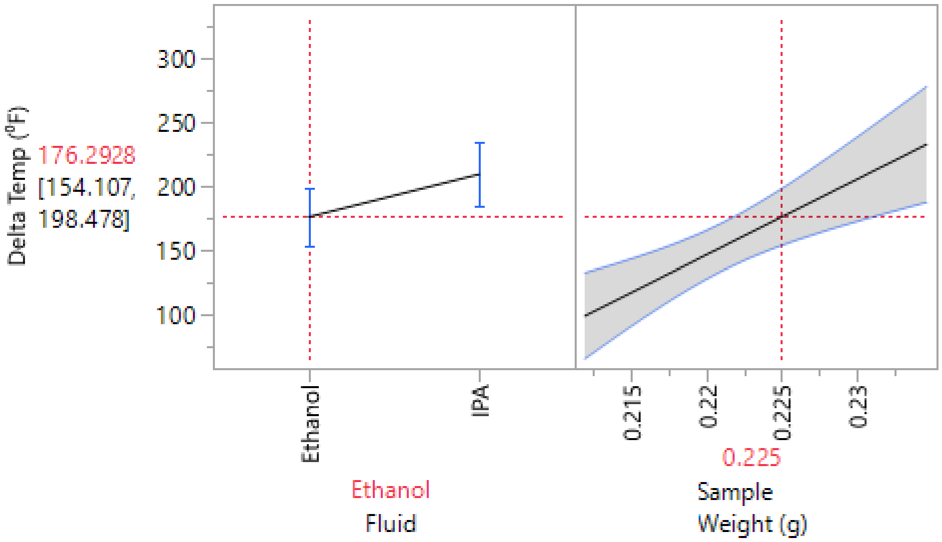


Figure 8.4.3-2. Prediction Profiler of Statistical Model

In conclusion, statistical tests and models were generated to determine significance and describe test data. Main effects were used for this analysis, and no higher-order effects were found to be significant. AIT was a function of fluid. Change in temperature was a function of fluid and sample weight. Change in pressure was a function of fluid, sample weight, and test pressure.

8.5 Conclusions

A test campaign was completed to determine the AITs of IPA and ethanol in gaseous oxygen at 10.3 and 15.2 MPa (1,500 and 2,200 psi). using special volatile material handling and test procedures. Results were obtained for five tests at each pressure. The data were analyzed statistically and delivered to the appropriate stakeholders.

9.0 Findings, Observations, and NESC Recommendations

9.1 Findings

The following findings were identified:

F-1. The average AITs for IPA in gaseous oxygen at 10.3 MP (1,500 psi) and 15.2 MPa (2,200) psi were 199.3 °C (390.8 °F) and 201.6 °C (394.8 °F), respectively.

F-2. The average AITs for ethanol in gaseous oxygen at 10.3 MPa (1,500 psi) and 15.2 MPa (2,200 psi) were 193.2 °C (379.8 °F) and 198.2 °C (388.8 °F), respectively.

F-3. The IPA autoignition delta pressure rise associated with autoignition increases with mass (i.e., a 2.5-time mass increase produces a ~4-time delta pressure rise).

9.2 Observations

The following observations were identified:

O-1. The average AIT for ethanol is close to that for IPA.

O-2. The delta pressure rise associated with autoignition is greater for IPA than for ethanol.

9.3 NESC Recommendation

The following NESC recommendation was identified and is directed Agencywide:

R-1. NASA should update the appropriate reference documents for oxygen and flammability to include the IPA and ethanol AIT data generated during this assessment.

10.0 Alternative Viewpoint(s)

No alternative viewpoints were identified during the course of this assessment by the NESC team or the NRB quorum.

11.0 Other Deliverables

No unique hardware, software, or data packages, outside those contained in this report, were disseminated to other parties outside this assessment.

12.0 Lessons Learned

No lessons learned were identified during the course of this assessment.

13.0 Recommendations for NASA Standards and Specifications

No recommendations for NASA standards and specifications were identified as a result of this assessment.

14.0 Definition of Terms

Finding	A relevant factual conclusion and/or issue that is within the assessment scope and that the team has rigorously based on data from their independent analyses, tests, inspections, and/or reviews of technical documentation.

Observation	A noteworthy fact, issue, and/or risk, which may not be directly within the assessment scope, but could generate a separate issue or concern if not addressed. Alternatively, an observation can be a positive acknowledgement of a Center/Program/Project/Organization's operational structure, tools, and/or support provided.
Recommendation	A proposed measurable stakeholder action directly supported by specific Finding(s) and/or Observation(s) that will correct or mitigate an identified issue or risk.

15.0 Acronyms and Nomenclature List

ACS	American Chemical Society
AIT	Autogenous Ignition (or Autoignition) Temperature
ASTM	American Society for Testing and Materials
°C	Degrees Celsius
CCP	Commercial Crew Program
°F	Degrees Fahrenheit
g	gram
IPA	Isopropyl Alcohol
J	joules
K	kelvin
kg	kilograms
kPa	kilopascal
mg	milligram
ml	milliliter
mm	millimeter
mol	moles
MPa	megapascals
MSFC	Marshall Space Flight Center
NESC	NASA Engineering and Safety Center
psi	pounds force per square inch
psia	pounds force per square inch absolute
WSTF	White Sands Test Facility

16.0 References

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2. *CRC Handbook of Chemistry and Physics*, 82^{nd} Edition, D. Lide, Ed. in Chief, 2001–2002, pp. 15–17.

3. Wegener, W., Binder, C., Hengstenberg, P., Herrmann, K.-P., and Weinert, D., "Tests to Evaluate the Suitability of Materials for Oxygen Service," Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986, D. W. Schroll, Ed., ASTM, Philadelphia, 1988, pp. 268-278.

4. JMP, Version 14.3.0., SAS Institute Inc., Cary, NC, 1989-2019.

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14. ABSTRACT

As part of a recent liquid rocket engine shutdown investigation, a commercial partner requested that NASA provide the autogenous ignition (or autoignition) temperature (AIT) of isopropyl alcohol (IPA). NASA provided the available data, but the data were somewhat scattered, likely due to test configuration and test technique differences. NASA Engineering and Safety Center (NESC) support was requested to experimentally determine the AIT of IPA and ethanol, both of which are extensively applied to propulsion systems. This report contains the outcome of the NESC assessment.

15. SUBJECT TERMS

Autoignition Temperature; Isopropyl Alcohol; Commercial Crew Program; NASA Engineering and Safety Center

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