

# Development of Reduced Toxicity Hypergolic Propellants

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**Hypergolic storable high-performance propellants fulfill a wide variety of mission roles in launch vehicle and spacecraft propulsion. The current favored storable hypergolic bipropellant combination of nitrogen tetroxide and monomethylhydrazine has significant handling and environmental issues due to their toxicity. Research into reduced toxicity storable hypergolic fuels has pointed to several types of reduced-toxicity fuel formulations which energetically react with hydrogen peroxide and provide similar performance to the current benchmark propellants. The authors have investigated the feasibility of multiple fuels to achieve adequate performance for bipropellant rocket applications with a hydrogen peroxide oxidizer. Results to date identify four promising candidates as potential competitors for the current benchmark hypergolic propellants.**

## I. Introduction

AMONG the existing liquid rocket engines, those using liquid oxygen/liquid hydrogen and monomethylhydrazine/nitrogen tetroxide are the best performing propellant combinations in the cryogenic and hypergolic liquid propellant categories, respectively. Specifically among the hypergolic propellants, monomethylhydrazine (MMH) represents the state-of-the-art fuel while Nitrogen Tetroxide (NTO), Mixed Oxides of Nitrogen (MON) & Inhibited Red Fuming Nitric Acid (IRFNA) are the most prevalent oxidizers. The aforementioned propellants have excellent performance characteristics in terms of specific impulse, density impulse, ignition delays and reliability. The MMH/NTO combination has been successfully used in the space shuttle orbital maneuvering systems (OMS) and the Reaction Control Systems (RCS). With the enormous increase in space activities since the 70's, certain inherent risks with the use of the aforementioned hypergolic propellants have increasingly become matters of concern as the propellants are highly toxic and difficult to handle. Hydrazine based fuels are also identified as carcinogens. Most of the oxidizers mentioned are highly corrosive. Storing highly toxic propellants onboard for long duration space missions poses a major safety hazard. With increased use, ground handling of these propellants creates workplace safety issues. For these reasons, there is a strong research interest in finding hypergolic propellants with far lesser toxicity and comparable performance with the state-of-the-art.

The present study focuses primarily on the development and testing of reduced toxicity hypergolic propellants whose performance approaches that of MMH/NTO. Key characteristics of desirable propellants are ease of handling & storability, low ignition delays, restartability and to an extent, low cost. Small ignition delays are likewise an important factor in avoiding hard starts in a hypergolic engine. Several previous efforts in the development of low

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toxicity propellants identified and successfully demonstrated the use of Rocket Grade Hydrogen Peroxide (RGHP) as a suitable oxidizer. Significant efforts include a class of fuels called Non Toxic Hypergolic Miscible Fuels, which are hypergolic with RGHP, developed at the Naval Air Warfare Center Weapons Division (NAWCWD), China Lake, California in 1999.

Due to hydrogen peroxide's inherent ease of handling, favorable physical properties such as high density and extensive experience in its handling at Purdue University, it was selected as the oxidizer of choice for this investigation. 90% RGHP was easily available at a reasonable cost. It was now necessary to develop a fuel hypergolic with hydrogen peroxide. There are many fuels compatible with hydrogen peroxide<sup>1 2 3</sup>, but most are not hypergolic. An attempt was undertaken to render a fuel hypergolic with hydrogen peroxide by adding a catalyst or a promoter.

The fuel is comprised of an additive (catalyst or promoter) dissolved in the liquid fuel to form a stable solution. There are two classes of hypergolic fuels based on the behavior of the additive. If the additive does not take part in the combustion process directly and only decomposes hydrogen peroxide, it can be classed as a catalyst and such fuels are known as catalytic hypergolic propellants. In catalytic hypergolic propellants, the heat released by the decomposition reaction of the catalyst and the oxidizer increases the temperature of the fuel. The fuel ignites at its auto-ignition temperature and the flame is sustained by the subsequent release of heat due to the combustion of the fuel and the decomposed oxidizer. Alternatively, if the additive actively takes part in the ignition process, it can be classed as a promoter and such fuels are known as reactive hypergolic propellants<sup>4</sup>. Ignition in reactive hypergolic propellants is achieved by the high exothermic reaction between the promoter and the oxidizer. The heat thus released, vaporizes the fuel which sustains combustion. In catalytic and reactive hypergolic propellants, the additive is primarily responsible for initiating combustion that renders the fuel hypergolic. It is also possible to have a fuel component which is actively hypergolic with the oxidizer along with the additive.

This investigation developed reactive hypergolic propellants. Reactive hypergolic propellants have lower ignition delays when compared with catalytic hypergolic propellants and will have a lower tendency to hard start. Low ignition delay allows for the use of shorter combustion chambers thereby conserving space and weight. Hence, the primary aim of this research was to develop storable reactive fuel solutions hypergolic with hydrogen peroxide with low ignition delay. In addition, both the components of the fuel solution, the additive and the liquid fuel were required to be low in toxicity.

## **II. Experimental Approach**

### **A. Development of fuel solutions**

As previously noted, the fuel type to be developed was reactive hypergolic. In this study, fuel solution is referred to as the combination of promoter and solvent. It was first necessary to identify suitable promoters that are known to react with hydrogen peroxide upon contact. The most natural are those of transition metal compounds. Previous research on these compounds by A. Musker, G. Roberts et al<sup>5</sup> identified compounds of iron, copper and manganese as possible catalysts for the decomposition of hydrogen peroxide. Although in that study, the compounds were used as catalysts (much like the decomposition of hydrogen peroxide using silver meshes), in this study, transition metal compounds were dissolved in organic solvents to form hypergolic propellants.

Compounds of iron and manganese have been considered in this study. Manganese in the form of Manganese Acetate Tetrahydrate (MAT) had previously displayed very good decomposition rate of hydrogen peroxide in Block 0 (Methanol + MAT), but it behaves as a catalytic hypergolic propellant. Therefore, it was decided to try different manganese compounds for this study. Chlorides of Manganese (II), Iron (II) and Iron (III) were considered for testing. Light metal – transition metal compounds like permanganates (sodium permanganate and potassium permanganate) were considered and were later dropped from testing since they do not dissolve in organic solvents to form a reactive hypergolic fuel solution. Permanganates exhibited rapid decomposition and have been demonstrated as a potential catalytic hypergolic propellants by A. Musker, G Roberts et al. Research in organometallics by T. L. Pourpoint & J. J. Rusek<sup>6</sup> prompted a fundamental study to see if any of these fuels were hypergolic with hydrogen peroxide. Among the organometallics, Ferrocene and methylcyclopentadienyl manganese tricarbonyl (MMT) were selected for examination. Finally, hydrogen rich metal hydrides were considered for testing as they are some of the most exothermic reducing agents of hydrogen peroxide.

Organic solvents have been considered primarily to dissolve the promoters. Suitable characteristics required of the solvents include high density, low vapor pressure and high heat of combustion. A literature review<sup>7 8</sup> indicated potential solvents to dissolve the selected promoters. Promoters were required to form stable solutions with the selected solvents and not precipitate out. Alcohols were the most common solvents for the transition metal compounds and the organometallic compound Ferrocene. Suitable solvents for the metal hydrides were identified. Sodium borohydride was the metal hydride of choice considering its lower toxicity compared to lithium or beryllium compounds, despite the fact that lithium hydride, lithium aluminum hydride and beryllium hydride are more energetic than sodium borohydride. Solvents needed to dissolve sodium borohydride needed to be polar aprotic. Pyridine, Dimethyl Formamide, Dimethyl Sulfoxide, Triethylene glycol Dimethyl ether (Triglyme) and Tetraethylene glycol Dimethyl ether (Tetraglyme) were identified as possible solvents with low toxicity for dissolving sodium borohydride. Table 1 displays the various promoter-solvent combinations that were identified for ignition delay testing.

**Table 1: Promoter-Solvent combinations**

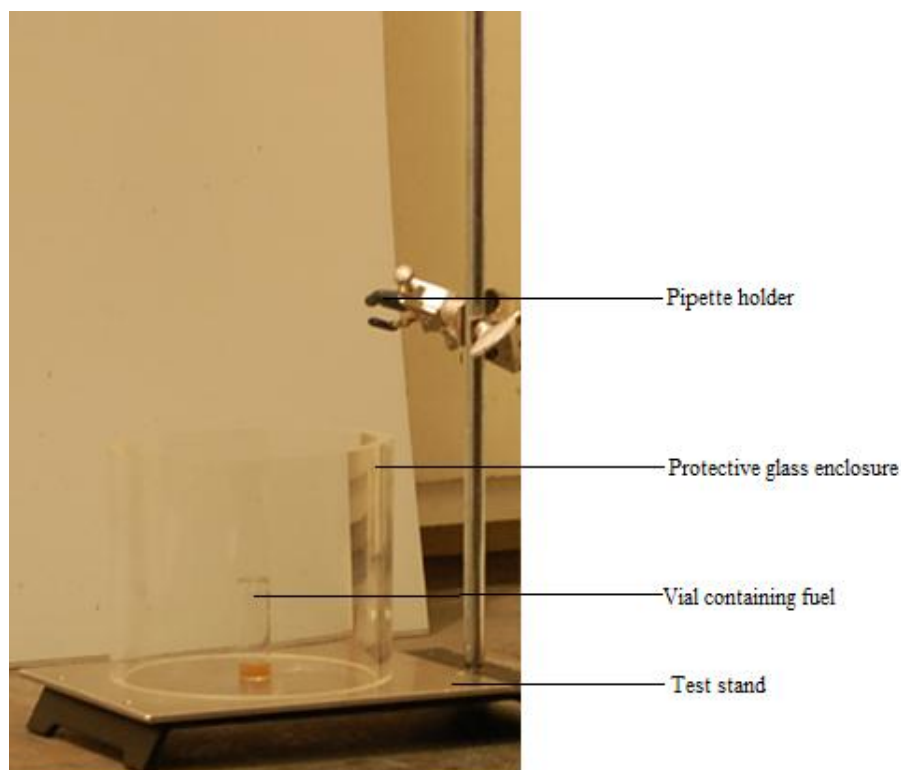
<b>Fuel Formulations</b>	
<b>Promoter</b>	<b>Solvent</b>
Sodium Borohydride	Dimethyl Sulfoxide
Sodium Borohydride	Dimethyl Formamide
Sodium Borohydride	Triethylene glycol dimethyl ether (triglyme)
Sodium Borohydride	Tetraethylene glycol dimethyl ether (tetraglyme)
Sodium Borohydride	Pyridine
Ferrous Chloride	Methanol
Ferrous Chloride	Ethanol
Ferrous Chloride	N-Butanol
Ferrous Chloride	Acetone
Ferric Chloride	Methanol
Ferric Chloride	Ethanol
Ferric Chloride	N-Butanol
Manganese Chloride	Methanol
Ferrocene	Ethanol
Ferrocene	Toluene
Sodium Nitrate	Methanol

### III. Ignition Delay Experiments

To provide a method for screening the large number of candidate fuels, drop tests were conducted. Hypergolic ignition can typically be observed by dropping a pre determined amount of oxidizer into a pool of fuel or vice-versa. In this study, the oxidizer was always dropped on the fuel with a fuel to oxidizer mass ratio of approximately 10.

#### A. Apparatus

All tests were conducted inside a fume hood at standard conditions. The ignition event was captured by a Phantom v7.1 high speed camera with a Sigma 24-70 f2.8 lens recording at 2000 frames per second allowing for a 0.5 millisecond temporal resolution. Hypergolic ignition delay was identified as the time lapse between the oxidizer droplet hitting the pool of fuel and the first occurrence of flame. The actual test setup comprised of a vial of fuel placed on a test stand with a pipette holder inside a protective glass enclosure as shown in figure 1.



**Figure 1. Experimental setup for drop tests.**

## **B. Procedure**

Initially, the oxidizer concentration was measured using a refractometer. Then the oxidizer was drawn into a pipette and clamped in the pipette holder with a drop height of 6 in. Fuels were mixed 1-3 days before drop testing. The containers were visually inspected for precipitation before transferring the fuel into the vial for testing. It is highly desirable that a component of the fuel formulation not precipitate out as this could cause injector and feed line clogging apart from rendering large ignition delays. The high speed camera was focused on the vial containing the fuel solution. One drop of oxidizer was allowed to fall from the pipette into the vial containing fuel to observe the ignition event. The high speed camera recording was manually initiated before the oxidizer was pushed through the pipette. Atmospheric conditions were recorded after every test. After the reaction, the test stand was prepared for the next test.

## **C. Results**

The summary of the drop test ignition delay results are shown in Table 2. Only those fuel formulations that displayed ignition are noted in the table. Signs of promoter precipitation in the fuel formulations were recorded. Promoter loading is based on the saturation percentage of that promoter in the solvent and is not an absolute value of the complete fuel formulation. The actual promoter loading is based on the total weight of the fuel formulation and not as a percentage of its saturation. The room temperature was measured using an off-the-shelf electronic thermometer placed inside the fume hood. Due to the sheer number of fuel formulations conceived for testing, only two drop tests were conducted for each fuel combination initially to identify potentially high performing fuels. Fuels that displayed promising results were further drop tested extensively. Impinging jet tests for measuring the ignition delay were conducted for some of the high performing fuels and have been detailed in the next section.

**Table 2. Drop test results**

<b>Fuel</b>	<b>Promoter Saturation in solvent</b>	<b>Actual promoter loading (w/w)</b>	<b>Precipitate observation (Y/N)</b>	<b>Oxidizer Concentration (%)</b>	<b>Avg. Ignition delay (ms)</b>
Sodium Borohydride + Triglyme	12% w/w at 20 C	8%	N	88.5	10.6
Sodium Borohydride + Tetraglyme	15% w/w at 20 C	10%	N	88.5	12
Sodium Borohydride + Dimethyl Formamide	18% w/w at 20C	12%	N	88.5	9.12
Sodium Borohydride + Dimethyl Sulfoxide	Unknown	1%	N	87.4	No ignition
Sodium Borohydride + Dimethyl Sulfoxide	Unknown	5%	Y	87.4	96.5
Sodium Borohydride + Dimethyl Sulfoxide	Unknown	10%	N	87.4	14*

\* Ignition observed but high speed camera was not triggered. Hence the delay was not an average but a single data point.

Ignition was not observed for all of the fuels under consideration. Only the fuels listed in table 2 demonstrated repeatable ignition. All ignition events were accompanied by loud pops. Ignition delays for these fuels were measured again using an impinging jet apparatus where the fuels were expected to exhibit delays much shorter due to better mixing of the fuel and oxidizer streams.

Hotfire testing of these fuels using a scaled injector and combustion chamber demonstrated the viability of the fuels in a rocket engine. The fuel combinations in the aforementioned table did not precipitate out when mixed in larger quantities for hotfires with a combustion chamber. Although sodium borohydride dissolved in pyridine displayed low ignition times, it was dropped from further testing due to possible acute toxicity issues with the solvent. Figure 2 shows a sample ignition event for fuel – sodium borohydride + Tetraglyme combination.

Most formulations involving transition metal compounds displayed various degrees of reaction rates but exhibited no ignition. It can be inferred that the promoter decomposes the oxidizer causing an insufficient temperature rise for the solvent to ignite. Both the organometallic compounds did not display ignition. MMT is currently used in the aviation industry to increase the performance of hydrocarbon based fuels and is added in small quantities. It could also be used to improve the performance of many of the organic fuels used in this study. No formal study has been undertaken to include the aspects of performance improvement by adding MMT. Promoter loading did not seem to have a particular trend in ignition delay times among the fuels that displayed ignition.

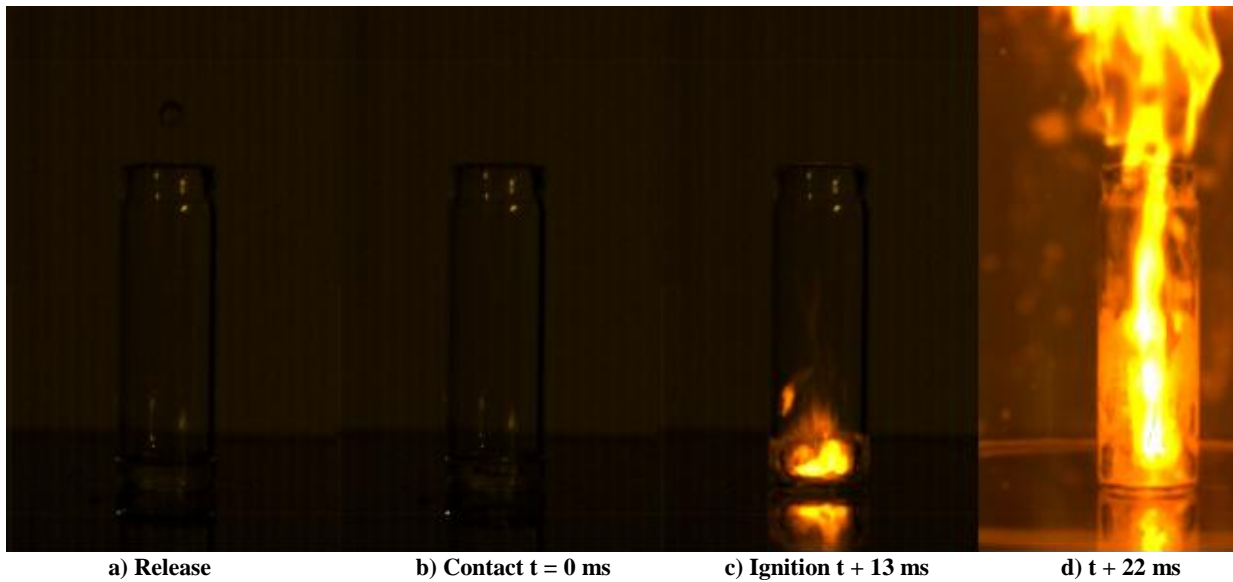


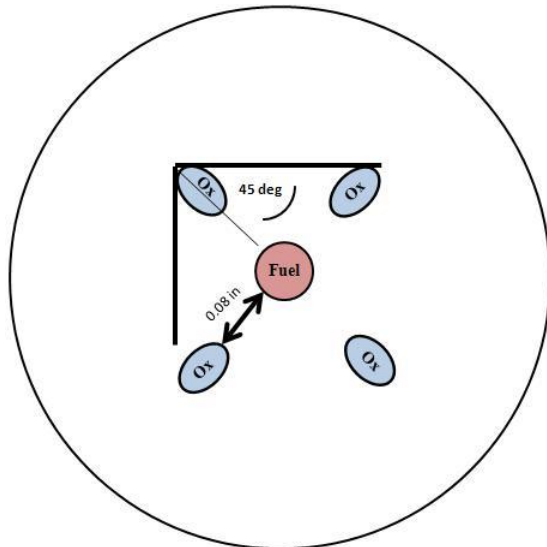
Figure 2. Typical drop test sequence. Timing indicated below photographs.

Higher promoter loading increased the reaction rates among those fuel formulations that did not display ignition but only displayed reactions. The percentage of sodium borohydride in the solvents – Triglyme, Tetraglyme, Dimethyl Sulfoxide and Dimethyl Formamide did not display any particular trend in the ignition delay times.

#### IV. Bipropellant Rocket Injector Experiments

##### A. Apparatus

A single-element rocket thruster was used to investigate the performance of the propellants under conditions similar to those in a full-scale propulsion system. The thruster was designed based on scaled data from whole-engine calculations for an engine with a vacuum thrust of 500  $\text{lb}_f$  and a chamber pressure of 250 psi. The single-element test bed was designed at to develop 60  $\text{lb}_f$  of perfectly-expanded vacuum thrust and achieve similar chamber pressure. A simple converging nozzle was installed on the thruster and subsequent reduced thrust levels were not measured.

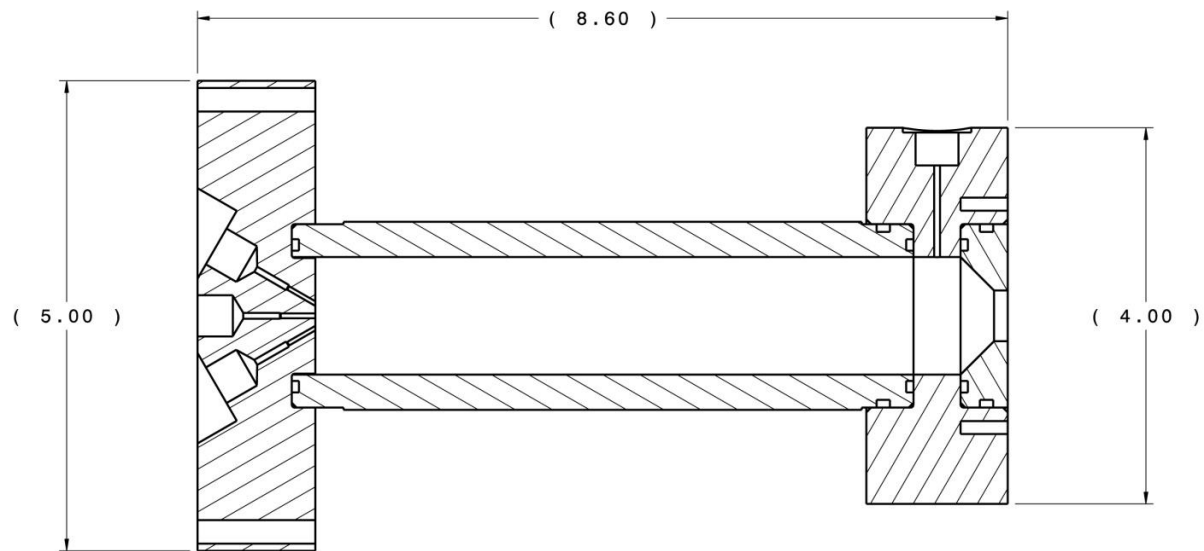


**Figure 3. Injector faceplate orifice positioning.** Bottom view illustrating positioning and type of injector orifices. Stream impingement angle of 60 degrees not shown..

The full-scale engine design was based on idealized rocket equations, mixing criteria from NASA-SP8089 Gill, G. S., and Nurick, W. H.<sup>9</sup>, Rupe mixing predictions, Rupe, J. H.<sup>10</sup>, and predicted propellant performance based on thermochemical software analysis<sup>11</sup>. The injector type, orifice diameter, and number were determined through trade studies comparing theoretical peak performance to hardware construction constraints. The optimal full-scale engine design included 8 injector elements with a pentad design. The pentad injector elements include a single fuel orifice at the center with four oxidizer orifices as shown in figure 3. The oxidizer stream total impingement angle was 60 degrees as illustrated by the angle of channels in figure 4. Oxidizer impingement upon the fuel stream occurred at half the total impingement angle or 30 degrees.

Figure 4 illustrates the test article design. The thruster was designed to emulate full-scale engine performance within constraints imposed by hardware and test stand limitations. The injector consisted of a solid stainless steel 304 faceplate

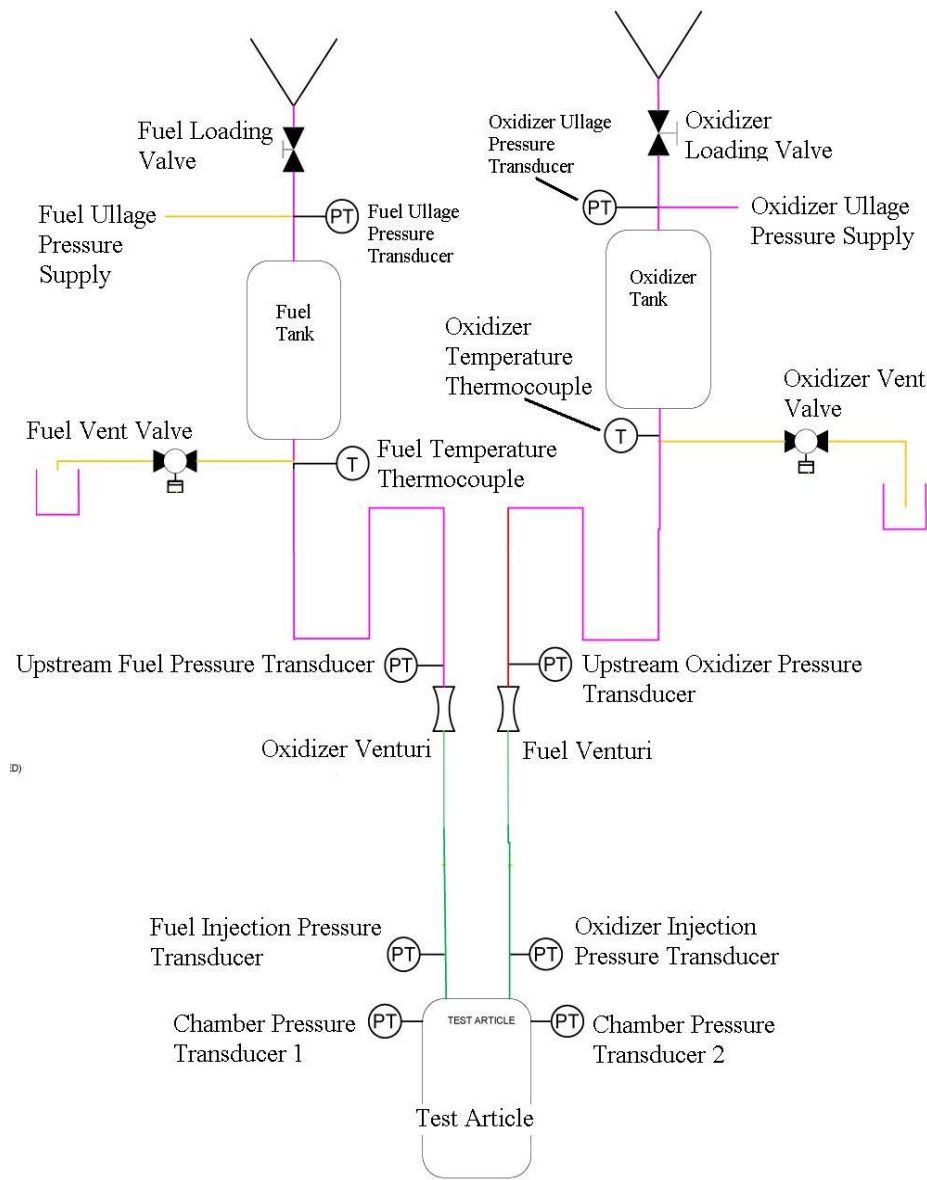
with holes drilled for a single pentad fuel and oxidizer injector element. It was noted after manufacture that the injector orifices were visibly misaligned, however cold-flow tests showed acceptable impingement. The thruster combustion chamber wall was cold-rolled steel AISI 1010 of sufficient thickness to withstand the anticipated thermal and pressure loads. Hot gas escape paths were sealed with viton o-rings. An instrumentation element at the end of the chamber, near the nozzle, was constructed from solid round stock carbon steel AISI 1026 drilled and channeled for two pressure transducers positioned 90-degrees from each other. A cold-rolled steel AISI 1010 nozzle element was affixed to the instrumentation element with threaded bolts designed to fail at an overpressure of 1000 psi. The elements were held together with ¼ inch threaded rod and bolted to the test stand in a vertical configuration as shown in figure A.1 in the appendix.



**Figure 4. Bi-propellant single-element thruster.** *Cutaway side view including all elements of the thruster. All dimensions in inches.*

The throat diameter was sized to develop a chamber pressure of 250psi based on ideal mixing and propellant performance. The chamber length was shortened from an ideal mixing length to 7in due to anticipated full-scale engine constraints. The thruster was designed to operate for a maximum of 4s, constrained by the thermal properties of the materials used. The nozzle was a simple chamfered converging orifice and not calculated to produce significant thrust.

The propellant delivery equipment and engine mounting test stand were of substantial construction and designed to deliver sufficient propellants for maximum hotfire duration of 2s. Propellant supply lines were of stainless steel construction throughout with the exception of brass fittings on the fuel side. Propellants were fed to the thruster using pressurized nitrogen. Propellant flow rate control was implemented through the installation of appropriately-sized cavitating venturies on fuel and oxidizer supply lines and the regulation of ullage pressures. Propellant supply and thrust chamber pressures were measured using Druck PMP 1260 industrial pressure transmitters with operating pressure ranges from 500 - 3000psi. Fuel and oxidizer supply temperatures were measured using Omega K-type thermocouples positioned below the propellant supply tanks. Figure 5 illustrates the relevant sections of testing apparatus plumbing and instrumentation.



**Figure 5. Test stand plumbing and instrumentation diagram..**

## **B. Procedure**

Impingement timing was determined during cold flow tests using de-ionized water. Impingement timing and mixing fan shape was recorded by a Phantom v7.1 high speed camera.

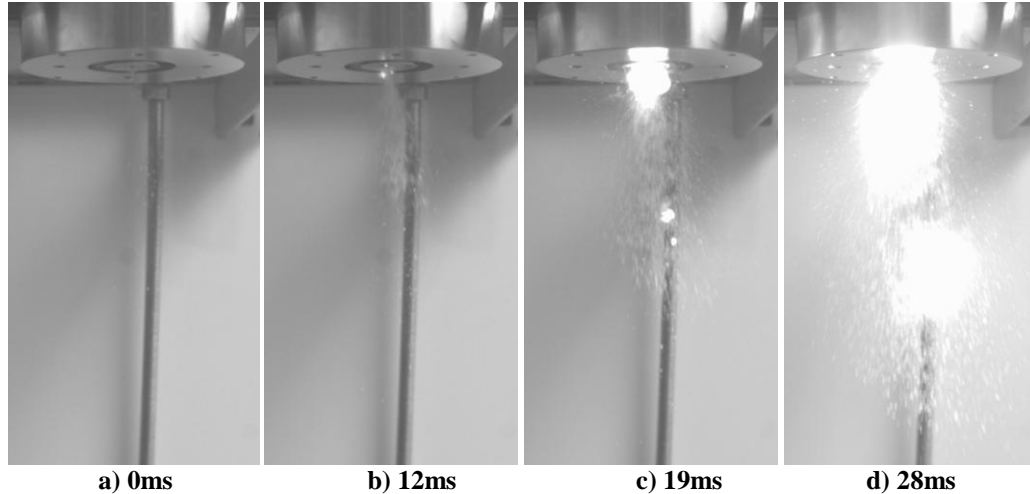
Variable ambient temperatures necessitated the addition of heaters to the fuel and oxidizer lines to maintain common test conditions across all propellant combinations. Two types of hotfire tests were conducted; Chamber – off tests were 0.75s hotfires conducted with all hardware removed except for the bare faceplate. Fuel and oxidizer streams intersected and reacted in the open air. The purpose of these tests was to investigate ignition delay times and observe ignition patterns via the high-speed camera. Chamber-on tests were 0.75-1.0s hotfires conducted with the thruster fully assembled to collect chamber pressure data and estimate propellant performance.



During the hotfire tests, the fuel stream was injected first with oxidizer streams impinging within 1ms. Supply system propellant pressures, temperatures, and ignition delays were recorded during chamber-off testing. Analysis of high-speed camera data was used to determine ignition delays for the propellant combinations tested. Supply system pressures and temperatures were recorded during chamber-on testing as well as chamber pressure within the thruster itself.

### C. Results

Single-element chamber-off thruster tests provided ignition delay times in good agreement with drop test values. Ignition delays for the four propellant combinations tested are displayed in table 3. Figure 7 shows still frames from the high speed camera illustrating the initial stages of combustion and ignition delay time.



**Figure 7. Typical Chamber-Off Ignition Delay Test.** Sequence of high-speed camera images detailing initial stages of combustion of sodium borohydride + Triglyme and RGHP during the third chamber-off hotfire. a) 0 ms - initial impingement of fuel and oxidizer streams. b) 12ms - first evidence of ignition near the injector faceplate. c) 19 ms - first evidence of ignition in the spray fan. d) 28ms - combustion spreads through majority of spray fan.

Table 3 catalogues the ignition delay times for each propellant combination tested in the thruster assembly.

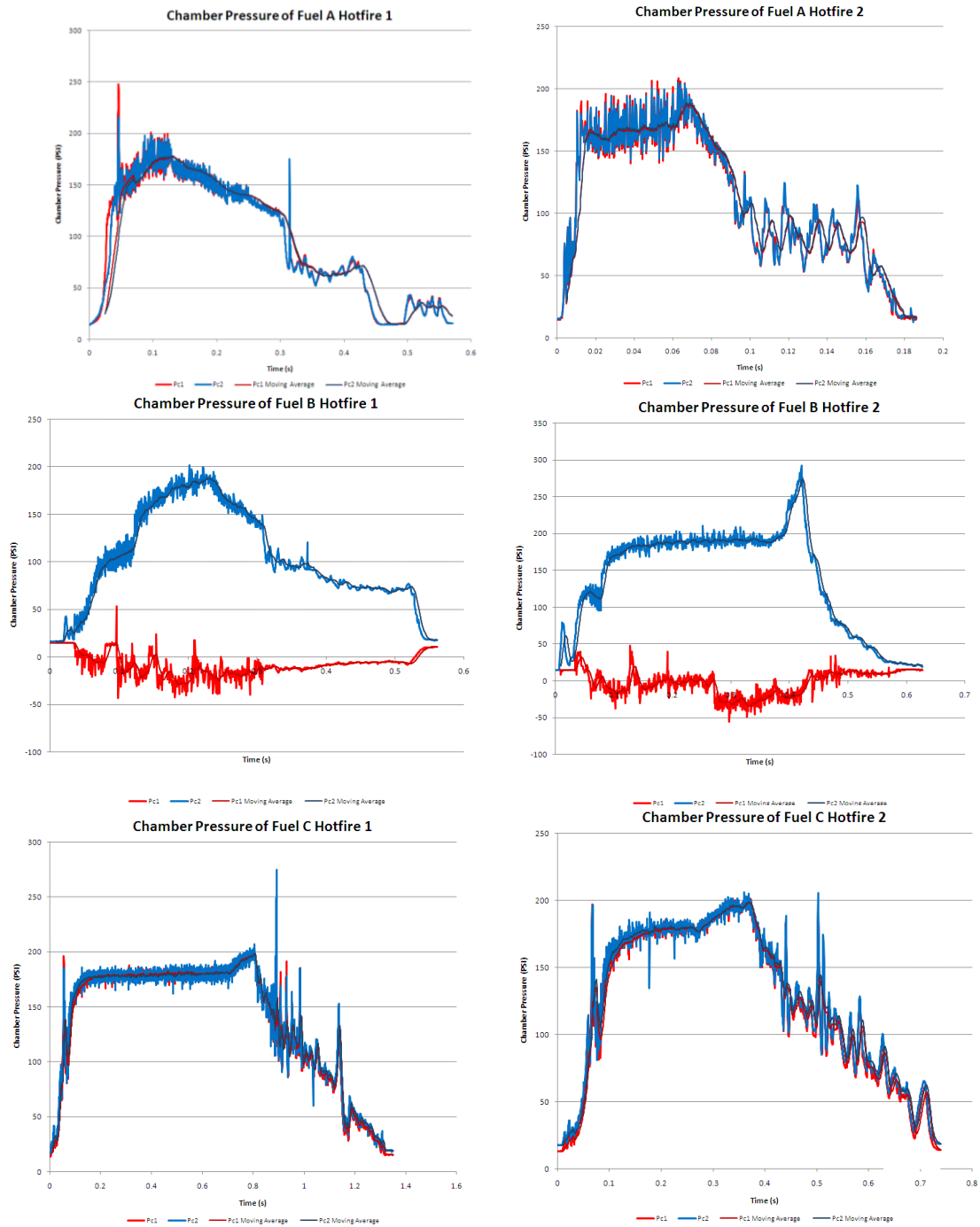
**Table 3. Ignition delay times.**

Fuel	Average Ignition Delay (ms)
Sodium Borohydride + Triglyme	9
Sodium Borohydride + Dimethyl Formamide	7
Sodium Borohydride + Dimethyl Sulfoxide	7
Sodium Borohydride + Tetraglyme	12

Pressure data from the single-element thruster during chamber-on hotfires indicated the development of steady-state flow within the approximate 1s run time of the tests. Pressures recorded for all propellant combinations were consistently lower than calculated values based on ideal mixing and sizing conditions. The lower chamber pressures may be a result of the deliberate shortening of the combustion chamber and the incidental misalignment of the injector orifices, leading to incomplete mixing and the subsequent development of less than full ideal chamber

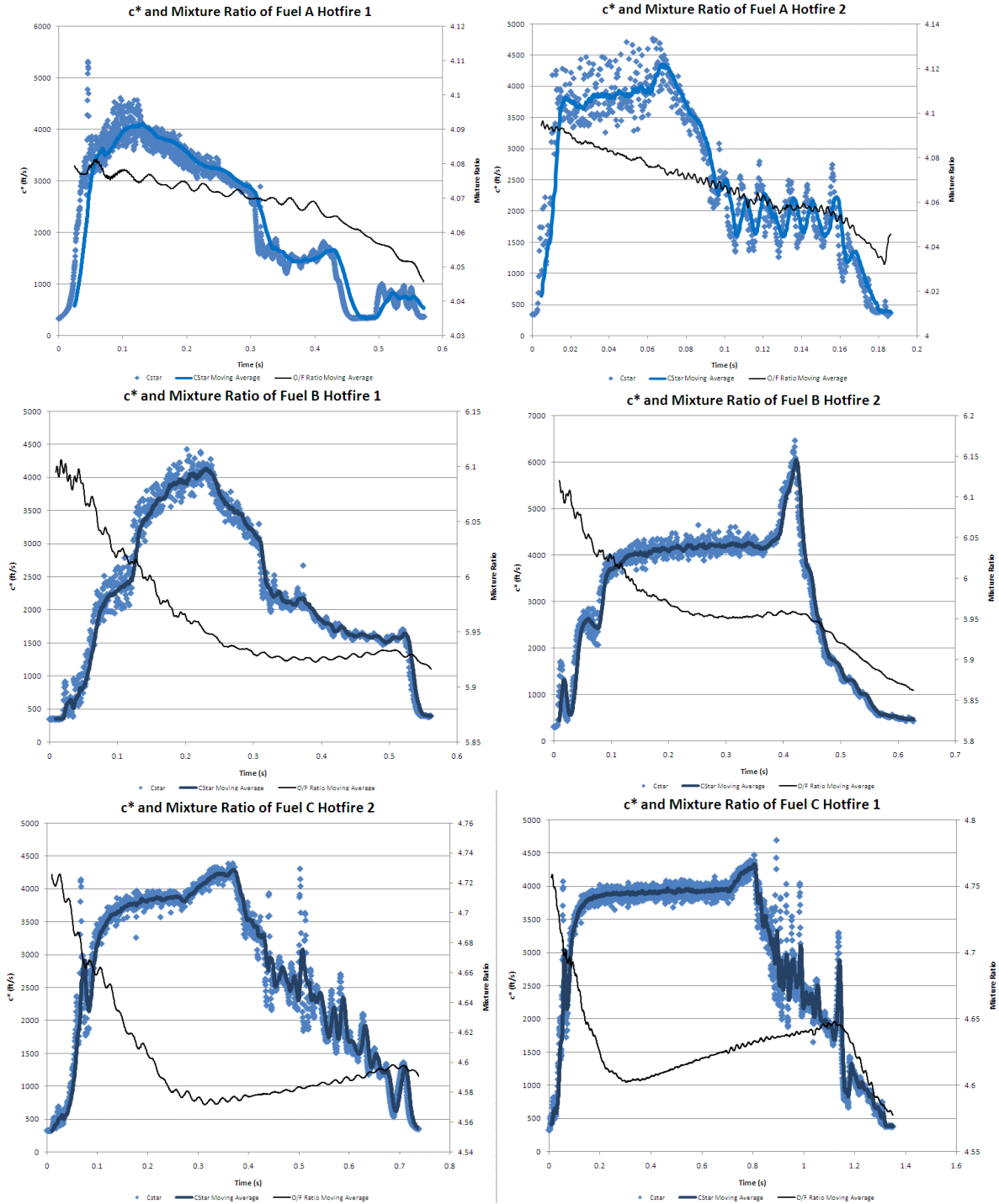
pressure. Post-test reduction of pressure and temperature data indicates off-nominal mixture ratios existed during testing, likely due to turbulence in the propellant supply lines.

Figure 8 displays chamber pressure data for a typical chamber-on hotfire of each fuel as well as a time-averaged line smoothed to  $1/100^{\text{th}}$  of a second per data point. Pressure data indicates that representative steady-state pressures were reached for the three fuel combinations tested - sodium borohydride + Triglyme (fuel A), sodium borohydride + Dimethyl Formamide (fuel B) and sodium borohydride + Dimethyl Sulfoxide (fuel C).



**Figure 8. Chamber-on Pressure Data vs Time.** Chamber pressure data and time-averaged lines are displayed for fuel A (top), B (middle), and C (bottom). The first test of each fuel is displayed on the left and the second test is displayed on the right.

Figure 9 gives the  $c^*$  and mixture ratio data of the reduced data while Table 4 summarizes the results of the hotfires. Due to off-mixture ratio operation, performance data does not clearly indicate a superior fuel combination between A, B, and C. Mixture B is the highest performing, but also operated at the highest mixture ratio. Optimal mixture ratios for all combinations were near 6. The reduced test data does indicate that the fuel combinations yield performance reasonably close to calculated values and within the range of  $c^*$  values usually found in bi-propellants used for rocket propulsion applications.



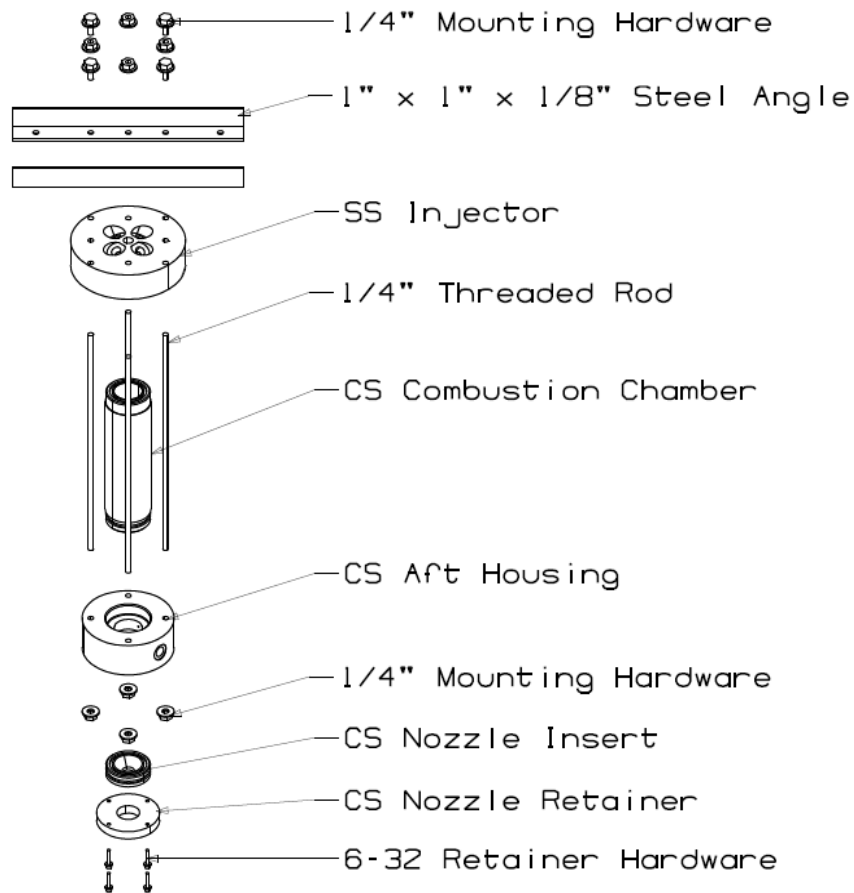
**Figure 9. Chamber-on  $c^*$  vs. Time.**  $c^*$  data and mixture ratio are displayed for fuel A (top), B (middle), and C (bottom). The first test of each fuel is displayed on the left and the second test is displayed on the right.

**Table 4. Performance Measurements.**

Fuel	Average c-star (ft/s)	c-star Standard Deviation	Average MR
A	3901	280 ft/s	4.08
B	4081	150 ft/s	5.96
C	3880	75 ft/s	4.6

Though instrumentation and flow quality problems prevented the hotfire testing from directly defining the highest performance propellant combination, the results from the tests are encouraging. The performance of the reduced-toxicity hypergolic fuel/oxidizer combinations in the test bed thruster is clearly within the range of typical rocket engines using toxic propellants. This indicates that all three propellants tested, with usable results, have the potential to be used as replacements to the current standard hypergolic fuels.

### Appendix



**Figure A.1 Thruster hardware.** Exploded view of test article and mounting hardware in vertical testing configuration.

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