



Hybrid safety

Introduction

“Rocketry success demands perfection...the first to feel this awesome imperative for perfection are the propulsion systems, especially the engines. Unless they operate flawlessly first, none of the other vehicle systems will get a chance to perform.” Noted professional rocketeer.

While there is no doubt that hybrids are considerably safer than traditional solid or liquid rocket engines, there is a popular misconception that they are totally safe, and never go boom. Sadly, an ill-designed hybrid - or one that has been subject to mechanical abuse - will most certainly go bang. Several have, and lives have been lost.

The following is a guide to some of the issues concerning the safe design and operation of amateur hybrids, focusing on the ‘green’ (non-toxic) oxidisers: nitrous oxide, liquid oxygen, and high-test (high-strength) hydrogen peroxide.

Note that Aspirespace has no hands-on experience with hydrogen peroxide as yet.

Needless to say, in these days when uncurbed litigation is the biggest threat to democracy and the pursuit of happiness, we have to state that the following is solely our opinion, and we accept no liability whatsoever for accidents arising from following the advice herein, nor do we assume liability due to errors contained within this paper.

This edition has greatly benefitted from reference 1, which is highly recommended, and from the good folks on the Arocket rocketry forum.

Items in **bold** are referenced in the glossary at the end of this paper.



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Cleanliness is next to godliness

The secret to the safe use of rocketry oxidisers is a hospital Operating-theatre level of cleanliness. The oxidiser shouldn't encounter even the tiniest trace of hydrocarbons until it is safely within the combustion chamber. If not, bad things happen.

What is the upper level of realistic cleaning effort required? There isn't one!

Humans and oxidisers

Rocketry-strength oxidisers regard you as a big fat-candle. You burn extremely well and you cannot be extinguished.

The Obituary Test (Ref. 16)

This is a test devised by the Arocket amateur rocketry fraternity to pre-assess rocketry safety, and should be your guiding star. Is what you're about to do going to lead to your Obituary reading: "What a Pratt!" rather than "These things happen in the pursuit of scientific advancement..."

For example, is a full oxygen cleaning of a **run-tank** regulator required? Tearing down, cleaning, and rebuilding the regulator is a serious chore.

This is a perfect application for the Obituary Test. If you didn't clean the regulator to oxygen standard and it killed you, would your ghost feel like an idiot? If the answer is yes, then clean the damn regulator to oxygen cleanliness standard!

What is a hybrid?

A hybrid - as the name suggests - is an engine that shares components from both solid-propellant and liquid-propellant rocket design.

A tank supplies liquid or gaseous oxidiser into a combustion chamber that contains a solid fuel; the term here literally meaning fuel only. (In a 'reverse hybrid' system, a liquid fuel is injected, and a solid oxidiser is in the chamber.)

The hybrid we're all familiar with is the log fire: the rate at which the solid fuel burns depends upon the draught of gas passing over the surface of the wood, which you can increase by opening-up the flue on the chimney, to increase performance.

In a typical hybrid, the oxidiser flows down a hole, or 'port' running down the centre of a thick-walled pipe of solid propellant fuel.

Once the hybrid has been ignited, some of the hot combusted gas is carried by the port flow to the surface of the solid grain where its heat is transferred into the top layer of this fuel by forced convection.

The heat then melts and vaporises a layer off of the exposed surface, which then gets carried off into the port flow, where it burns in a rather interesting way; it actually burns in a narrow zone within the boundary layer known as the flame zone or flame sheet, which is at a depth between the fuel-grain below and the oxidiser flow above that is close enough to stoichiometric (best mix) to support combustion.



Part 1: oxidiser choice

The propellants used in hybrid motors encompass all the familiar ones, plus some that may be new to you.

Though in today's world, the choice of oxidisers are increasingly being restricted to the more environmentally friendly ones: oxygen, High-test peroxide, and nitrous oxide.

As far as rocketry performance is concerned, hydrogen peroxide or just raw oxygen give better **Specific impulse** and **Density Impulse** (packageability) than nitrous oxide ('nitrous'), but nitrous is so much more user-friendly than these, which is why it's so popular.

The United Kingdom Rocketry Association's safety code (version 5, section 5.2 gives some excellent advice on nitrous hybrid usage and safety. (www.ukra.org.uk)

One of the challenges in educating people about the dangers associated with oxidisers is their prior perceptions: oxygen is used in hospitals to make you better, nitrous is used by dentists, peroxide is used as a hair dye. These can lull you into a false sense of security when you begin your hybrid rocketry, but beware. Oxidisers can bite! Having said that, they can be handled safely provided you take all necessary precautions. I'll try to outline many of these precautions in this article.

First off, nitrous oxide is not highly reactive to humans like hydrogen peroxide, nor excessively cold or excessively shock-sensitive like liquid oxygen (Lox).

Nitrous is, however, a powerful anaesthetic and is used as such by the medical profession: inhale too much and you might never wake up again.

Inhale Lox or peroxide vapour, and say goodbye to your lungs: terminal frostbite or combustion.

You can carry bottles of nitrous in the back of vans (but not in cars, there should be a barrier between driver and bottle in case of a leak as nitrous is a powerful anaesthetic; stupidly we've all done it, but keep all windows open.) Unless you want to be flame-grilled in an accident, you'd be crazy to carry Lox or peroxide in your own vehicle; they have to be pre-delivered to a proper test-site or launch-site which adds to the cost.

This table outlines some performance pros and cons to consider when choosing between oxidisers for hybrid rocketry applications. Safety issues will be discussed presently.

	Liquid oxygen	High Test Peroxide	Nitrous oxide
Composition	O ₂	H ₂ O ₂	N ₂ O
Vapour-pressure	Low: needs pumped	Low: needs pumped	55 Bar at room temperature: self-pressurising
Typical oxidiser to fuel ratio	2.5:1	6:1	7:1
Density (kg/m³)	1140	1414	Around 800 (liquid phase)
Storage	Cryogenic (finite boil-off rate)	Chemically reactive (passivation required)	Inert (anaesthetic)
Flame temperature	High: One-shot graphite nozzles	Moderate: graphite nozzles can be reused.	Moderate: graphite nozzles can be reused many times.
Vacuum ISP with plastic fuel (seconds)	Highest (315)	Middle (290)	Lowest (230)
Mixture sensitivity	High	Low	Low
Stored just below the Critical temperature?	No, colder	No, colder	Yes

Vapour pressure:

Liquid oxygen and peroxide require external pressurisation; either from a separate gas supply or by using a pump. In contrast, nitrous oxide self-pressurises to a respectable 55 bar at room temperature.

Typical mixture ratio:

Liquid oxygen has a much more equal mixture ratio than peroxide or nitrous but this means that a Lox hybrid's fuel grain is disproportionately larger than the other two, which have smaller fuel-grains but larger oxidiser tanks.

Plastic fuel grains tend to end up long and thin, giving long, thin, and so structurally inefficient combustion chambers. This mitigates against single-port Lox-plastic hybrids.

Density:

Nitrous suffers here, requiring large, heavier pressurised tanks.

Storage:

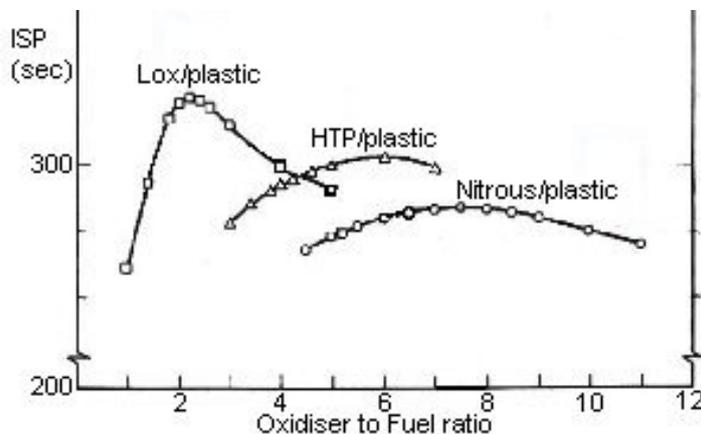
The Scaled Composites WhiteKnight carrier aircraft took around an hour to climb to altitude and then release SpaceshipOne: if its hybrid had used Lox, a lot of it could have evaporated by then.

Flame temperature:

The combustion chamber temperature of a Lox hybrid is a lot higher, causing materials problems.

Mixture ratio sensitivity:

Lox is much more sensitive to the wander of oxidiser to fuel ratio (mixture ratio) that occurs during a hybrid burn as this graph of **Specific impulse** (ISP) shows:



In many ways, nitrous oxide behaves as a slightly feebler, but user-friendlier peroxide; in fact the technologies developed for peroxide rocket engines are applicable, including catalytic decomposition.

Peroxide itself is a popular hybrid choice; Richard Brown developed a very successful man-rated peroxide hybrid for his attempt on the motorcycle land-speed record, and currently the Bloodhound SST supersonic car is due to be powered by peroxide hybrids.

Storing/filling oxidisers

Store **fill tanks** out in the open, and keep all plumbing above ground to aid leak detection. Have a source of water nearby for firefighting/deluge/dilution.



Cleanliness

...is next to godliness as they say. For oxidiser systems this is imperative, as most contaminants are at the very least fuels, and often will cause an explosive compound with oxidisers. Use special degreased tools, and only use those tools for the oxidiser system alone. Use lint-free cloths, and avoid creating metal particles/swarf.

Training

The safe use of rocketry oxidisers is all about education. The rocket firing team must all be trained to handle the oxidiser being used, understand its chemistry, its materials compatibility and non-compatibilities (potential to go boom with certain materials), and be aware of all safety implications. Getting them all to read this document is a good start, and then a safety document (Standard Operating Procedures, SOP) specific to your hybrid engine needs to be written and read.

The SOP should include:

- Notification of the designated safety authority during hazardous operations (The site Safety officer).
- Protection of personnel.
- Prevention and detection of oxidiser leaks.
- Elimination of ignition sources.

The design of safe facilities and equipment should consider human capabilities and the limitations of personnel responsible for operations: your team are probably volunteers.

The firing team need to demonstrate proficiency in the use and care of protective equipment and clothing and safety equipment, and also proficiency in maintaining a clean system and clean equipment in oxidiser service

Hazards analysis

Perform a hazards analysis on the hybrid engine system, particularly the oxidiser components:

- Determine the most severe operating conditions (pressure, temperature, flow velocity, rubbing of components).
- Evaluate the flammability of the plumbing materials when in use. Test unusual materials for flammability.
- Evaluate potential unwanted ignition sources: flammable materials can only be used in sections of the plumbing where there is absolutely no chance of ignition.
- Be cautious of single-barrier failures: a single-barrier failure is a leak in which only the primary containment structure is breached. Such a leak introduces oxidiser into a region not normally exposed to oxidiser. The materials or configuration of parts in this region may not be compatible with oxidiser!
- Single-barrier failures are often overlooked, but the potential for component-part failures - such as diaphragm failures - can cause hazardous oxidiser-enriched environments, and can cause a substantially increased risk of ignition near electrical components for example.
- Attempting to correct single-point failures simply through procedural actions is not a reliable method. [A single point (of) failure is a part of a system that, if it fails, will stop the entire system from working. These are undesirable in any system with the goal of reliability]. Design the system safe instead.
- Include hazards associated with contamination (muck, dust).
- Review secondary hazards, such as seal leakage to electrical equipment.
- Design equipment so that power losses, control pressure leakage, or other loss of actuation sources, return the equipment to a fail-safe position to protect people and property in the event of an accident.

Fire

One of the main hazards of oxidisers is their ability to create a fire. All three sides of the triangle must be present: a fuel, the oxidiser, and an ignition source. But with rocketry oxidisers, the fuel could even be metal pipework, and the ignition source a tiny spark from static electricity, friction, or simply dropping something heavy onto a pool of oxidiser soaked into the ground (which is then the fuel).

The key to the proper use of rocketry oxidisers is the use of compatible materials and the elimination of ignition hazards.

Humans and their clothing burn really well when splashed with oxidiser:



You have been warned! (the picture is of a cotton shirt splashed with Lox).



Part 2: pressure vessel and plumbing mechanical safety

Manual versus remote fill as it affects propellant tanks

Any container that can safely withstand the pressure without bursting will do for a propellant tank.

Stand next to a vessel pressurised full of high-pressure gas or vapour however, and your delicate bits are right next to a potential grenade; it's the shrapnel of the exploding casing of a grenade that does the ripping through everything bit.

The United Kingdom Rocketry Association (www.ukra.org.uk) quite rightly regards standing anywhere near a cylinder under pressure as potentially hazardous.

Their safety code only allows people to be close enough for them to manually fill a tank if the tank is deliberately designed with a high margin of safety to prevent bursting. For experimental hybrids UKRA will rightly require sufficient proof of tests performed to verify the tank safety if anyone is to be near it.

Remote-filled systems allow the luxury of smaller tank safety margins, and so thinner-walled, lighter tanks, but the potential mass saving if using an ultra-thin tank is proportionately smaller as the vehicle diameter gets smaller. For example, small HPR aluminium tanks suffer minimum gauge problems, and over-proportionally large bolt-holes which raise stress.

If nobody's ever going to be near a tank while it's pressurised, i.e. only remote filling and dumping throughout the whole life of the tank, then the safety-factor on burst can be low to keep the tank mass down. 1.5 times the expected pressure is a reasonable remote-fill margin for a reusable tank, but pressurise the tank and plumbing a few times to this pressure before first flight to be sure.

Obviously, you *can* safely be close to the massively over-strengthened fill-tanks used to transport Nitrous from the suppliers; they're properly engineered and manufactured to allow you to move the contents around the country after all, and they're tough unless you drop them on their necks and break the valve off. Then you get a rocket alright: the tanks can fly at very high speed and cause major damage. Such a commercial container is safe enough to handle (though not idiot-proof) but is far too heavy to fly.

Commercial hybrids are available such as the Aerotech manually-filled nitrous hybrid system which has this kind of strengthened tank, but for experimental hybrids UKRA will rightly require sufficient proof of tests performed to verify the tank safety if anyone is to be near it. Remote-controlled filling of flight tanks is preferred.

It doesn't really need saying, but any homemade tank from a rocket that has suffered a recovery-system failure needs re-tested.

Aspirespace's smaller vehicles (FLARE, ADV2a, and ADV2c/Rickrock) utilise manual fill.

Remote-filled systems allow the luxury of smaller tank safety margins, and so thinner-walled, lighter tanks, but the potential mass saving if using an ultra-thin tank is proportionately smaller as the vehicle diameter gets smaller.

For example, the number of fabric layers of a composite tank is an integer, it can't be a fractional number, so small composite tanks can't be made with an optimum composite thickness; they're over-heavy.

Similarly, small aluminium tanks suffer minimum gauge problems, and over-proportionally large bolt-holes which raise stress.



Electroformed (grown metal) metal tanks can be made exceedingly thin, but annoyingly the electroformed metals are often incompatible with oxidisers (okay for Lox, all incompatible for nitrous).

A manual fill system is simpler to construct, but involves a higher level of design and testing of all tanks and plumbing that will be pressurised; safety of the filling crew is the prime consideration.

Obviously, you can safely be close to the massively over-strengthened tanks used to transport nitrous or Gox from the suppliers; they're properly engineered and manufactured to allow you to move the contents around the country after all, and they're tough unless you drop them on their necks and break the valve off. Then you get a rocket alright: seriously, the tanks can fly at very high speed and cause serious damage.

Such a commercial container is safe enough to handle (though not idiot-proof) but is far too heavy to fly.

Thin-wall aluminium tanks

So just how strong do propellant tanks have to be to be reasonably safe to be near without being too heavy to launch?

Military missile-design literature recommends a design safety factor of at least 2.0 times expected internal pressure on pressure-vessels that are to be manually handled while they're pressurised.

But this applies to a production tank; the fundamental point is that a batch of military production tanks have been tested to destruction to ensure that they really do burst at well over the 2.0 safety factor, and not a worrying 2.00001

Now we amateur groups don't tend to make batches of tanks therefore we can't perform a suitable number of destruct tests.

Several rocketeers over the decades, making one-off tanks, have learnt to their cost that home-made thin-walled tanks, especially aluminium ones, need a burst safety-factor of at least three to handle the inevitable dings, scratches, and accidental alteration of the metal properties (typically inadvertent annealing) during manufacture.

Unexpected stress raisers can cause ally to rip apart at much lower average stresses than you bargained for, especially if you try welding the 6000 T series aluminiums, as this locally kills their heat treatment which is what originally made them strong. They must be re-tempered.

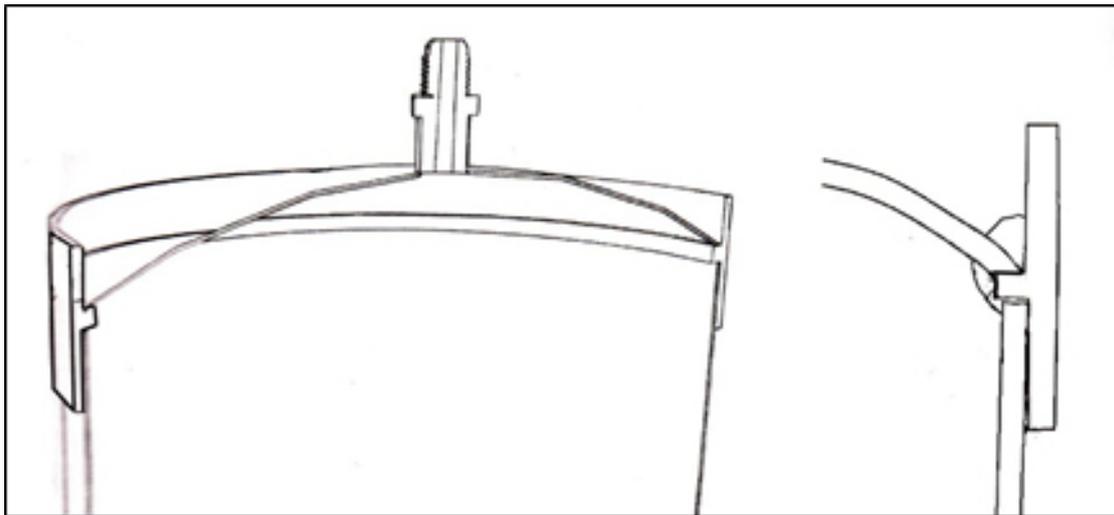
So for manual filling, then Aspirespace's approach, based on the personal experiences and advice from engineers who make home-made tanks, is that you should test your hybrid's one-off tanks to ensure that they safely withstand three times the expected pressure. For nitrous oxide this would be three times the vapour-pressure of nitrous on a hot day without failure, or 3 times 60 Bar = 180 Bar.

CO2 paintball-gun tanks, for example, are designed to be carried around during such adult tig-tag, bashed off trees, and people have sometimes fallen on them, without injury. These tanks are all pre-tested to around five times the expected internal pressure (of CO2, which has a higher vapour-pressure than nitrous), and are stamped to say as much; so a bad batch will fail during factory testing well before reaching the shops.

So Aspirespace have been comfortable enough with manual filling of nitrous hybrids that use such paintball tanks, such as our highly successful FLARE and ADV2 vehicles.

Paintball tanks are a good safe choice, but they're somewhat overheavy. Also, most (Luxfer) paintball tanks use an imperial UNF internal thread on their necks; sourcing a suitable tap can be tricky.

DIY aluminium thin-wall tanks are often formed from end-domes and cylinders welded together. The 6000 series aluminiums are usually heat-tempered (designation 'T') and when welded lose this temper and so lose considerable strength. The tank therefore needs a post-weld heat treatment to restore the temper. The 2000 series aluminiums, though weaker, are not tempered so avoid this problem. Another way to get around this problem is to add extra metal around the joint: this lowers the stress in the metal so that the loss of strength isn't an issue. This strengthening band is shown here in cutaway, and forms a useful tank skirt for attachment to the rest of the vehicle:



Composite tanks

Several decades ago, space and aerospace companies identified the need for low mass pressure vessels. The composite overwrapped pressure vessel was developed to provide weight savings without a loss in safety margins over traditional tanks. The design consists of a thin liner of material, typically a metal, overwrapped with a continuous fibre yarn impregnated with a resin such as epoxy. The weight saving over a traditional tank is around 25 to 50 percent.

The general advice from composite manufacturers is that DIY home-made composite tanks have resulting properties that are far too variable from tank to tank to predict: use remote filling only.

More to the point, when composites are damaged or experience many pressurisation-depressurisation cycles, the ensuing micro-cracks are rarely visible on the surface but will grow under mechanical stress, so the tank may rupture the next time it's pressurised without any visible prior warning.

Even thin-wall aluminium liners are not thought to provide the whole structure with enough uniformity to allow anyone to be near one when pressurised.

One would hope - and industry demands - that the tank would leak before bursting, releasing the pressure. But this confidence only comes from very carefully controlled manufacturing, so that any cracks are known to be 'stable'. Stable can be defined as a crack that has a length less than 10 times the wall thickness of the vessel, and has a **stress intensity** less than the material toughness (Ref. 1).



Don't assume that a leaking liner makes the whole tank safe; a failed liner transfers more of the load to the composite, which can then undergo brittle failure. Make the composite strong enough to carry the entire pressure load.

If you treat your composite tank like eggs from the moment it's made, then throw it away after its first flight then maybe...

But the whole point of all the hassle of a composite tank is to go for absolute minimum tank mass, so there's absolutely no point in building a composite tank that isn't remote-filled.

Composite tanks need a separate internal liner because raw composite leaks the contents inside through microcracks, and furthermore the resin can cause nitrous oxide to 'explosively' decompose, or form an explosive with Lox and probably HTP.

Plastic liners are lightweight, but you must ensure that the plastic is one that isn't a fuel or catalyst: using a polythene liner for example turns the tank into a potential hybrid if blowback of chamber gasses upstream occurs! Use the fluoroplastics such as PTFE or FEP.

Composite tanks are very sensitive to impact: write an impact-mitigation work plan (no big spanners to be dropped onto the tank) and consider safety shields around the tank.

Tank testing

No pressure-vessel, no matter who or what company made it, is at all safe until it's been pressure-tested.

Until somebody's tested it, don't use it.

If there's no information stamped on its base, or if some rocketeer who you know is bad at recovery-systems sells you a scarred and pitted second-hand tank, regard it as dangerous until you've tested it.

Don't even assume that a tank specifically sold for nitrous hybrids will take full nitrous pressure; some are only designed to take the lower pressure that you get with a permanently-open vent-hole system.

Remember to test the tanks to the safety-margin that they'll need: at least 180 bar for any nitrous tanks you'll ever be near.

Many carbon dioxide fire extinguisher bottles are tough enough even to transport nitrous around in, but beware second-hand ones: test them first.

A safer testing method

The only safe (safer) way to test a high-pressure system is hydraulically: never use compressed gas or you're just making a gas-pressurised grenade again.

Buy a hydraulic hand-pump and some hydraulic jack-oil from an auto-store (such as Halfords or the like). Before pumping the tank up, make absolutely sure you've bled all the air out of the system (no bubbles) by separately filling the tank and pump completely full of oil before you connect them.

For safety, arrange to have at the very least a solid brick wall between you and the tank during testing, or preferably the corner of a building.

You don't need to be able to see the tank anyway: any leaks will show up as a steady drop in pressure on the hand-pump's pressure-gauge, their location revealed afterwards as a green stain on fresh newspaper.

And if the tank bursts, the loud splurt of a soggy explosion sounds exactly like an enormous aquajet/water rocket going off.



Bits of burst tank won't go very far under fluid pressure, because fluids can't store energy in compression nearly so well as gasses, but jack-oil is impressively messy: the whole area gets slimed.

Make sure nobody else can wander into the danger area while you're testing, and take extra precautions if you're testing a large tank.

After testing is finished, thoroughly clean the inside of the tank because jack oil is a fuel. Cleaning procedures are covered in later chapters.

The proof test

You're looking for proof that the tank won't rupture; hydraulically test the tank to 1.3 or 1.5 times the highest pressure you ever expect the system to encounter. (The proof test isn't a to-destruction test, so the safety factor is lower.)

Remote fill systems

Sadly, tanks that can safely withstand three times nitrous pressure can be too heavy for some vehicles. If this is the case, then you should resort to developing a safe remote-filling system.

If nobody's ever going to be near a tank while it's pressurised, i.e. only remote filling and dumping throughout the whole life of the tank, then the safety-factor on burst can be 1.0001, or however lucky your rocket is feeling.

1.5 times the expected pressure is a reasonable remote-fill margin for a reusable tank, but pressurise the tank and plumbing a few times to this pressure before first flight to be sure.

Pre-launch and aborts

Remote-fill systems must be designed to fail-safe, i.e. will empty the **run-tank** if electrical or pneumatic/hydraulic power is lost.

Otherwise - excepting a sniper - there's no way to depressurise a remote-fill tank if the remote-fill system dies and the hybrid didn't launch. You can't approach the pad without serious risk to yourself, so the pad could be out of bounds until the Lox or peroxide has all evaporated, or the nitrous pressure lowers the following winter!

The simplest approach for nitrous hybrids is the one commonly used; the vent-hole's secondary function is a deliberate leak to slowly lower the tank pressure in the event of an abort.

A deliberate leak is a good idea even on a vent-free system, and the time it takes to depressurize the nitrous tank is entirely up to you. (Evacuate the pad for an hour though, and the Range Safety Officer may become displeased!)

Below 20 Bar as read on a pressure-gauge through binoculars, gives the required safety factor of 3 again on the original 60 Bar nitrous pressure, and you can then approach the pad.

For safety, vent all remaining nitrous in an aborted fill, and wait 15 minutes before re-filling otherwise the tank will be excessively cold and thrust will be lost (see our 'Physics of nitrous oxide' article).

For Lox or peroxide systems externally pressurized by nitrogen or helium, a tiny leak at the top of the **run-tank** still works as a means to slowly depressurize the tank gas.

Launch

The remote-filling mechanism absolutely must disconnect from the rocket at or before launch, otherwise half the pad gets carried aloft and the rocket vehicle crashes. Following the advice of NASA literature on remote disconnection systems, consider backup mechanisms in case the primary disconnection mechanism jams.



Too many backups decrease reliability (the KISS principle) but one backup increases reliability greatly.

For example, if the tie-down strap on the commercial Hypertech system breaks or wasn't installed properly, then the backup is that the fill-line burns through almost instantly, before the rocket has left the pad.

The Reaction Research Society has launched rockets using an ingenious re-rig of a standard hydraulic quick-disconnect coupling: The coupling's release-collar is tied to a stake in the ground, and the male part is fixed into the rocket pointing aft. When the rocket pulls upward on the coupling at launch, the lanyard tied to the release-collar goes taut and the coupling disconnects automatically.

This is a good design for the backup release, which Aspire have adopted as ADV2b's backup disconnect.

Note that some quick-release couplings can't be disconnected before de-pressurising the line.

Hydraulic overpressure, the head space

Pick up any fresh bottle of camping-gas or CO₂ and give it a gentle shake; the sound of waves sloshing inside reveals that the bottle hasn't been completely filled with liquid, there's obviously a small amount of gas in there as well.

This small 'head space' or 'ullage' of gas is in there for a purpose, because after filling, the liquid's density will change with any future changes in temperature. In fact just-subcritical fluids like nitrous, carbon dioxide, or butane, can change density rather a lot with temperature if they're around room temperature. Similarly, Lox will change density as it warms.

The danger is that if the temperature increases, the liquid density will drop (see our physics of nitrous oxide article).

If the tank was completely full of liquid, then the tank's fixed volume now won't be enough to contain the mass of liquid as it expands. (i.e. increases its volume) If the tank is stoppered, the liquid will then self-pressurise. Liquids don't compress easily, so the ensuing hydraulic self-pressure can often be enough to burst the tank, or any closed-off feed plumbing downstream of the tank.

To prevent such an accidental hydraulic overpressure, then just as in nature's design of the egg, a small percentage of the tank volume is deliberately left free of liquid to allow for expansion with temperature. This gas pocket can then compress to absorb reasonable volume changes without overpressuring the tank.

(Seriously large **run-tanks** benefit from either a **pressure relief-valve**, which is a commercial safety valve designed to open at a set over-pressure, or a commercial **burst-disc** that is designed to burst at a set over-pressure.)

For nitrous, this 'head space' of gas is often created by situating a vent-pipe a little way below the top of the tank, so that the liquid never fills above the level of the vent (see our 'The physics of nitrous oxide' paper).

As every excess gram of run-tank matters, what is a reasonable minimum volume of head-space?

Using tables of nitrous properties with temperature (Ref. 2) the Aspirespace **run-tanks** are designed to absorb the liquid expansion caused by a 10 degree C increase in nitrous temperature after the vent is closed at the nominally British climate's 15 degrees C. This requires a 12% ullage (12% of the tank volume is vapour) which reduces to 0.7% at 25 degrees C.



In practice, we use 13% to 15% depending on how accurately we know the tank internal geometry. (See our 'Physics of nitrous oxide' article for the required calculation.)

The hybrid engine plumbing

Rigid pipework and welded joints are preferred for integrity, but swaged joints are better for modification or replacement of parts: cutting a pipe and welding it again can introduce metal particles and other contamination into the pipework.

Threaded joints should be avoided with oxidiser system plumbing, as the threads are difficult to keep clean. Where threaded joints can't be avoided, use PTFE tape to seal the thread, not sealing compound. Be careful not to let strands of PTFE come loose and fall into the pipework as they can block injector orifices or jam valves.

Be careful not to over-tighten stainless steel swaged fittings as they can 'pick-up' (cold-weld) i.e. jam solid.

Flexible hose should be used as little as possible and only where absolutely necessary, for example, where thermal contraction could occur.

Plumbing should be properly clamped and supported to minimise flexing, chafing, abrasion, resonance, and mechanical strain that could lead to the pipework breaking. The supports should allow thermal expansion or contraction where necessary. Rigid pipes should be supported as close as possible to each bend in the pipe.

Connectors and fittings that are to be disconnected during normal operation should be provided with tethered end-plates, caps, plugs, or covers to protect the plumbing from contamination or damage when not in use. Keep it clean!

Avoid rust coming into contact with nitrous vapour as it can act as a catalyst for the nitrous. This is particularly a problem with cheap zinc-passivated hydraulic fittings as their plating comes off or goes 'chalky' and the sulphide chemicals contaminating industrial-grade nitrous can rot them over time. Stick to stainless steel and aluminium fittings where possible.

Hybrid engines vibrate: use mechanical devices such as safety wire wrapping or bent metal tabs to prevent nuts unscrewing themselves.

When designing the plumbing, take note of high transient fluid pressures from pyrotechnic valves or other rapidly-opening valves that suddenly introduce high pressure fluid into a low-pressure pipe. The sudden 'jolt' can give pressures much higher than the pressure occurring once the system settles down.

To measure the tank and combustion chamber pressures you need to make a tiny hole in the tank and in the chamber. These holes (pressure-taps) are connected to very narrow pipes that lead to pressure sensors. Using only a small hole means that the flow won't be disturbed much by the presence of the hole.

Make the narrow pipes more than 20 centimetres long and running upwards with the sensor on top. Any longer and the gas trapped in the pipes won't be able to react quickly enough to sudden changes in flow pressure, but any shorter and the hot combustion chamber gas could get all the way up the pipe and melt the sensor.

Proof-test and leak-test the plumbing.

Trapped gas/vapour will tend to collect in the highest points of the plumbing. Consider a valve there to bleed this off as with your radiators at home.



Right way up!

When you test your nitrous hybrid, it's important to remember to mount the nitrous **run-tank** so that it's upright, as it will be in flight. The outlet of the tank must be at the bottom so that liquid nitrous comes out first and not vapour. Though this may seem obvious, it has been known for a static firing attempt to get this wrong.

The combustion chamber

As well as having suitable internal thermal insulation (such as Tufnol phenolic composite) the combustion chamber must not leak otherwise a plume of hot, escaping gas will erode the leak far wider and cause a burn-through, so leak-test the chamber by plugging the nozzle.

Viton rubber O-rings are sufficient to prevent leaks in nitrous engines, but they melt at lowish temperature so should be shielded from the hot chamber gas by a covering of insulation (this traps a pocket of cool air which protects the O-ring).

For Lox engines, use copper gaskets.

Of course, the chamber is also a pressure vessel so needs to be hydro-tested as was done with the tanks (see earlier).

Nitrous leaks

Leaks show up in any pipe-joints carrying the liquid phase of nitrous as regions covered in ice; the nitrous sucks heat out of the atmosphere as it leaks out to atmospheric pressure and vaporises (see our physics of nitrous oxide article), freezing the water-vapour in the air around the leak. It'll freeze your hands or face too if they're near a leak: wear goggles and gloves when you work with nitrous.

Cryopumping (sort of)

As the first syllable suggests, this pitfall should only occur at cryogenic temperatures such as when you use Lox, but it caused the demise of our first nitrous hybrid Rickrock flight (ADV2c), so is worth mentioning.

[Cryopumping occurs when the metal of some cavity is chilled by cold fluid in contact with it. The cold reduces the local pressure, and reduces the volume taken up by (increases the density), of the air in the cavity, and so more air flows into the cavity from outside.

Despite any previous purging of the cavity by inert gas, this air flowing in from outside contains water-vapour, which freezes on contact with the cold wall.]

Rickrock 1's manual-fill system used a manual vent-valve, this was a simple banjo-bolt that was screwed shut after filling.

On this occasion, we didn't realise that it had frozen partly open due to a phenomenon akin to cryopumping up the threads of the bolt during a pause in the filling operation. This caused a leak that slowly lowered the run-tank pressure, and Rickrock 1 sailed away on a low, flat trajectory that broke the recovery system due to too high an apogee airspeed.

Although the term 'cryo'-pumping implies cryogenic temperatures, vaporising nitrous will also chill metal components sufficiently for this to occur.

We've since redesigned the vent-valve for Rickrock 2.

Reverse flow

This is thought to be the main hazard to hybrids, the most likely hazard to occur:

Terminal flatulence

We all want the highest combustion chamber pressure to get the biggest **Specific impulse**; as close to the tank pressure as possible, but there's a practical limit.



A common trend is to use too large a hole/holes for the injector, in order to minimise the pressure-drop across it, but this is a very dangerous practice.

The pressure drop is there partly to prevent very hot combustion chamber gasses having the potential to flow back upstream into the feed system or tank if there is a chamber pressure pulse. Too low a pressure drop across the injector encourages such audible forward-reverse flow oscillations in the chamber:

Screaming/farting hybrids are NOT cool, they're dangerous: hot gasses could get back into the feed system and start a fire (or decompose the nitrous there in a nitrous hybrid).

Use a smaller orifice size to get the chamber oscillations under control.

Reference 3 - in line with most injector design practices - advises a pressure drop across the injector of 20 percent of the combustion chamber pressure at the end of the burn when the tank pressure is at its lowest.

On a particularly cold day in the UK (i.e. a low tank pressure), a commercial nitrous hybrid with too large an injector orifice (causing too low an injector pressure drop) went boom. The Space Propulsion Group have had several nitrous hybrid feed pipe explosions while still with liquid in the tank, believed to be caused by reverse flow into the feed system.

For example, at the end of the burn, our Aspirespace nitrous tanks are at about 36 Bar, so we tune the nozzle throat size so that the chamber pressure is no higher than 30 Bar at the end of the burn as 30 Bar times (100+20) percent = 36 Bar.

The injector pressure drop therefore has to be 36 - 30 = 6 Bar

To re-iterate, make sure that the upstream feed pipe (between **run-tank** and injector) is suitably wide enough (large enough cross-sectional area A_{pipe}) that the flow velocity within it is less than 10 metres/second (Ref.3, i.e. effectively stagnant) to minimise pressure losses in this pipe, otherwise you'll get an unwanted pressure drop along the pipe which reduces the injector pressure drop.

Then use the following equation to design the number of orifices required by the injector:

$$n = \frac{\dot{m}_{oxidiser}}{A_{orifice}} \sqrt{\frac{K}{2 \rho_{oxidiser} \Delta P}}$$

(from the continuity equation inserted into Bernoulli's equation)

Where n is the number of orifices, $\dot{m}_{oxidiser}$ is the oxidiser mass flow rate in kg/sec, $A_{orifice}$ is the internal cross-sectional area of one orifice (metres²), ρ is the liquid oxidiser density, and ΔP is the required injector pressure drop (Pascals) at the end of the burn.

We've experimentally determined the energy loss (head loss) constant K for nitrous oxide to be approximately 2.0 as the nitrous partially vaporises within a sharp-edged injector orifice giving a froth of 2-phase flow. (Length of the orifice divided by its diameter was around 4). For Lox or peroxide use a K value from the internet.

Another way to blow up the feed system is to let hot igniter gas back-flow through the injector. Don't let this happen.



Part 3: Nitrous oxide decomposition hazards

The day the hybrid world changed

In 2007 a terrible accident occurred at the test site of a very large hybrid. A large flight-weight **run-tank** of Nitrous exploded, and several Rocketeers died or were severely injured.

This accident caused a lot of soul-searching in the nitrous hybrid community, most of the work on nitrous hybrids stopped completely – both amateur and commercial. Many amateur rocketry groups became scared of nitrous and switched to a different oxidiser. It took more than a decade to calm everyone down again.

So what exactly happened?

The facts are that a large composite tank had been filled with Nitrous in preparation for a flow test of the hybrid injector. This test was cold: there was to be no ignition of the engine. However, a device was added downstream of the injector that was electrically powered.

During the flow test, this electrical device malfunctioned and reacted with the Nitrous flowing through it and exploded. This fired the injector rearwards into the composite tank which shattered. With the loss of the injector, a flame front would have travelled back into the tank.

The majority of the Nitrous spontaneously decomposed into nitrogen and oxygen gas, with a large release of heat from the decomposition (the casualties suffered severe burns) that raised the run-tank pressure. This over-pressurised the run-tank, and as there were no pressure relief devices on the tank such as a **burst-disc** or **pressure relief valve**, the tank burst.

Although a lot of damage then occurred to the test area, it was significantly less damage than would have occurred had there been a detonation of the Nitrous. The definition of a detonation is that the flame front spreads through the Nitrous at supersonic speed. In fact, no evidence has yet been uncovered to suggest that Nitrous will detonate.

The data suggests that spontaneous decomposition can occur, spreading through the Nitrous at a rapid pace, but significantly slower than a typical fuel/air deflagration.

It appears that only the Nitrous *vapour* is the culprit, liquid Nitrous doesn't appear to support continued decomposition because the surrounding liquid soaks the heat out of the reaction (this is called **quenching**): all attempts in the literature to ignite liquid Nitrous have failed.

It's probable that the damage was caused by a **BLEVE** event. This stands for Boiling Liquid Expanding Vapour Explosion.

When the Nitrous vapour decomposed and, along with the injector, ruptured the tank, the tank pressure suddenly dropped to the pressure of the atmosphere outside the tank. This caused all of the liquid Nitrous to flash-boil into vapour, expanding enormously in the process.

This expansion provided the energy for propagation of further cracks in the run-tank wall (shrapnelling) and then propulsion of these fragments of tank at very high velocity.

The expansion was further enhanced by a greater number of moles in the decomposed gasses and their increased compressibility factor (a so-called 'real' gas effect).

As this was happening, the now greatly increased amount of Nitrous vapour decomposed, causing a release of heat which caused further gas expansion.

Complete decomposition of the Nitrous in a run-tank will theoretically cause a twenty times increase in pressure, though the tank will have burst long before reaching that pressure.



[Note: Pressure relief valves are sized to release pressure fast enough to prevent the pressure from increasing beyond the strength of the vessel, but, for Nitrous, not so fast as to be the cause of an explosion. An appropriately sized relief valve will allow the liquid inside to boil slowly, maintaining a constant pressure in the vessel until all the liquid has boiled and the vessel empties. Basically, too large a relief valve orifice or burst diaphragm could cause a BLEVE event.]

Alternatively, there are suggestions that the run-tank was filled hours earlier then left to the mercy of the Sun: the test was then run at the hottest part of the day. The ambient temperature was reported as 40+ degrees C.

Nitrous goes supercritical at 36 degrees C (see our 'Physics of nitrous oxide' paper) so the tank or plumbing could've contained supercritical Nitrous. Supercritical Nitrous is very susceptible to pressure-shock which will result in a very high velocity decomposition during which temperatures can exceed 3,000 degrees C. This may also have caused the demise of the MARS team's Deimos Odyssey Nitrous hybrid in the heat of the Blackrock desert USA.

It turns out that Nitrous is a good solvent, particularly if it's supercritical. The run-tank used a composite tank liner, so bare composite resin was in contact with the Nitrous, as well as the waxy deposits that you get on the surface of composites. Nitrous can dissolve the hydrocarbon binders in the resin. This became the fuel, or possibly the catalyst for the decomposition. Plastic saturated with Nitrous can decompose 'explosively' when ignition energy is provided.

Static discharge

What provided the ignition energy?

It was a very hot, dry day in the test area that day, which is conducive to the build-up of static electricity. Our Nitrous hybrids typically have metal tanks and combustion chambers, so they're earthed when in contact with our metal test-stands. But this hybrid had a composite tank and chamber. Assuming that the injector plate was metal, then this could have built up a large static charge as the Nitrous flowed through it.

The electrical conductivity of Nitrous is low enough that with flowing Nitrous it is theoretically possible to produce a large enough static discharge to initiate decomposition.

As liquid Nitrous flows through an injector, Nitrous vapour appears. This vapour can be easily ignited by an electrical spark at typical tank pressures, and even a very feeble spark will do it (0.14 Joule). One rocketeer from the Arocket rocketry forum recounts: "I can tell you from personal experience that a big spark will set off Nitrous."

Electrical arc

The other candidate for ignition was the device attached downstream of the injector. It appears to have exploded, perhaps oil or grease plus electricity reacted with supercritical Nitrous.

Scale effect

Another issue with the hybrid was its very large size. The radius of the feed pipes of many small amateur hybrids are smaller than the **quenching distance**. This is a size (around 7 millimetres for pressurised Nitrous, though it changes with pressure) below which the metal walls of the pipe are near enough to soak up any rogue heat energy from decomposition, stopping the reaction dead.

In Ref. 9, the researchers could only get Nitrous vapour to sustain a reaction in a ¼ inch radius (6.35 millimetre radius) metal pipe by heating it prior to ignition (204 degrees C and 55 Bar). In a 1/2 inch radius pipe and larger, there was no quenching at typical Nitrous pressures.



Rocketry use of nitrous

Nitrous has many uses, from medical anaesthetic to dispensing whipped cream, and it's been used for over 100 years. There have been very few accidents. (Excepting the poor souls whose colons exploded during surgery due to catalytic decomposition of nitrous anaesthetic reacting with intestinal chemicals. Farted out of existence is a sad way to go...)

But the *rocketry* use of nitrous is generally a new thing, we're still in the learning phase:

- Rocket propulsion is unique in that large quantities of nitrous are stored at room temperature in thin-walled flight-weight tanks.
- The combustion chamber is closely coupled to the **run-tank**. This is a significant source for ignition which does not exist in other applications.
- Nitrous oxide can be safely handled in extreme conditions in the liquid state, but hazards exist in the vapour state or supercritical state at elevated temperatures and/or pressures.
- The release of thermal energy is by **exothermic** molecular decomposition; this has a handy side-effect in that it helps rocket engine combustion stability.
- There is a large energy release upon decomposition, about double that produced by an equivalent weight of TNT.
- Despite its potential decomposition hazard, if handled properly nitrous is one of the safest rocketry oxidisers.

Let's look at the hazards:

Contamination

The presence of even a small amount of fuel or catalyst material in tanks and feed plumbing can greatly reduce the energy threshold required for initiation of nitrous decomposition. A mixture of nitrous and 9% hydrocarbon (ethanol) - initially at only 40 degrees C - exploded in a lab.

I've listed compatible and non-compatible materials at the end of this chapter, though an awful lot of materials have still to be tested: if you google on nitrous material compatibility you'll find that most of the websites are way behind the current research.

The way to deal with contamination issues is to give the system a damn good clean; equipment must be thoroughly degreased before use.

Professional rocketeers working on manned nitrous hybrid systems have - in my view - been forced to go completely overboard on their cleaning since the aforementioned accident. They're using the strict procedures used for *oxygen* rocket systems.

If you're going to all the bother of full oxygen cleanliness, you might as well use liquid oxygen (Lox) in your hybrid as it'll give you a much higher **Specific impulse**.

Here's one of their 4-step cleaning procedures:

- Dust and loose contaminants are removed by scrubbing, then the piece of plumbing is cleaned with oxygen cleaner, then rinsed in water.
- Ultrasonic cleaning of the part.
- Rinse and soak in de-ionised water 3 times to remove the oxygen cleaning agent.
- Dry the parts with nitrogen.
- Inspect cleaned parts, re-clean if they fail:
- Visual inspection looking for loose contaminants or grease.
- Wipe with a white lint-free cloth (check that the cloth remains clean).
- Ultraviolet lamp inspection.
- Store all cleaned parts in sealed plastic bags and re-inspect just prior to assembly.

In my personal opinion, an adequate cleaning procedure for us amateur nitrous rocketeers is to wash your **run-tank** and feed plumbing in **trike** or chloroform, then acetone or Isopropyl alcohol, then rinse with good clear Highland water.



Bear in mind that thousands of grubby HPR nitrous hybrids get assembled in muddy fields and lobbed skyward without incident, though their small size might be saving them (quenching distance).

Incompatible grease

Although a properly installed O-ring in a properly designed groove will seal perfectly when dry, it's become an amateur and HPR rocketry habit to smear some grease around the O-ring, primarily to give a sacrificial heat-proof coating.

There have been several instances of people reaching for their solid-propellant rocketry supply of petroleum jelly/vaseline and smearing nitrous hybrid engine O-rings with it.

A little thought reveals that such grease is a fuel, whilst the nitrous is an oxidiser: sure enough their hybrids blew up upon ignition.

Use only oxidiser-compatible greases: these fluorocarbon greases can be bought from SCUBA diving suppliers or rocketry vendors.

Adiabatic compression

This is also called 'water hammer' (even though there's no water), and until recently we all thought it only occurred with pure high-pressure oxygen (Gox). Aspirespace had to design against it for our H2O Lox hybrid.

What happens is that if a run valve is opened suddenly, Gox rushes down the feed pipe until it hits another shut valve or obstruction such as the injector. The Gox's momentum piles it up against the shut valve, and the temperature rises purely because of the compression. This self-heating can reach the ignition temperature of the pipework, and start a fire.

In nitrous' case, the self-heating can reach its auto-decomposition temperature.

Here's a note from XCOR: "We had a few milligrams of fuel in a Snaptite valve which we didn't know about; we had some nitrous in it as well, and we hit it with a water hammer. Adiabatic heating made the fuel/nitrous mix diesel, and that was enough - in that small space - to set off the nitrous. The result was a very loud CRACK! and a thoroughly ruined Snaptite valve."

The way to prevent adiabatic compression is to avoid sudden rushes of oxidiser: open run valves slowly and reduce the dead-space downstream of valves. Dead volumes in the feed lines (e.g. a tee fitting) are prone to adiabatic compression. Reduce the pressurisation rate of your run-tank as you fill it to no more than 20 psi per second (Ref. 11) to avoid adiabatic compression. The way to do this is to reduce the diameter of both your fill pipe and your **run-tank vent**.

Bruno Berger at the Swiss Propulsion Laboratory (Ref. 13) understandably worries about imploding cavitation bubbles of nitrous vapour, because when they implode, they can generate very high temperatures.

As the liquid nitrous flows down the feed pipe between tank and injector its pressure drops, which causes some of the liquid to flash into vapour (two-phase flow) causing lots of little vapour bubbles.

If you get an adiabatic compression in the feed line or at the injector, these bubbles could implode. If there are enough of them imploding then the froth could generate decomposition energy faster than the liquid nitrous can quench it, leading to the feed pipe over-pressurising (blows up).

The way to minimise two-phase flow is to follow the advice of Ref. 3 which recommends keeping the pipework wide enough to keep the liquid nitrous flow-speed V below 10 metres per second, because the drop in pressure of the nitrous as it flows through a pipe varies with V^2 (Bernoulli's equation).

Use the mass continuity equation:

$$V = \frac{\dot{m}}{\rho A}$$

where \dot{m} is the mass flow rate of nitrous in kg/sec, and A is the cross-sectional area of the pipe in metres². Use a nitrous density ρ of 744 kg/m³ (a hot day)

Keeping the flow-speed low similarly avoids pressure changes due to sudden changes in pipe cross-section (particularly at valves) which can trap a pocket of vapour. Avoid changes in cross-section.

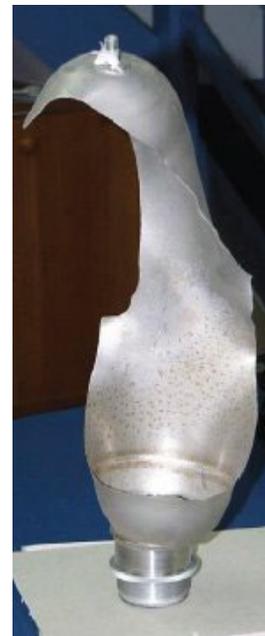
The Knights Arrow team avoid nitrous vapour entirely by pressurising their liquid nitrous **run-tank** using a nitrogen-powered piston. This raises the nitrous way above its vapour-pressure so that vapour cannot form. Personally, I think this is over-kill, but as it's a manned hybrid, it's their call.

Reverse flow: Flashback through nitrous vapour

Flashback of hot chamber gasses into the nitrous vapour *after* all the liquid has run out is thought to be a greater hazard (Ref.11) than before the liquid runs out. This is because there's much more vapour in the feed system, and this vapour goes all the way up and into the tank, so the tank can go boom too.

Hybrids have exploded when the liquid nitrous has run out, and a chamber pressure spike has reversed the flow.

Here's a picture of the remains of a **run-tank** (thanks to Troy from Arocket) after a flashback event at the end of a run resulted in an explosion of the vapour within the tank:



Inadvertent liquid engine, a hard start

One of the biggest bangs of recent years in the U.K. professional (defined as getting paid for it) rocketry field occurred when a nitrous hybrid engine accidentally became a liquid engine, and then blew up.

The nitrous must not be in liquid form once it's inside the combustion-chamber because of the danger of it pooling in corners of the engine; molten plastic fuel may be pooling in the same corner.

In this case, the nitrous was injected tangentially into the chamber instead of axially for several promising technical reasons, but centrifugal effects re-compressed the nitrous, and at the higher pressure it reverted into a liquid again. A design oversight allowed molten plastic to occupy the same area, and it did the classic liquid rocket engine 'hard start' upon ignition.

This bang was compounded by the fact that the feeble igniter was situated at the nozzle instead of where you ought to put it: close to the injector, so the chamber could happily fill with unburnt nitrous.

One way to fill the chamber with liquid is to use too large an injector orifice, with its resultant too low a pressure drop. This could keep the nitrous from vaporising properly.

Arocket have seen "a very scary explosion of a thin-walled steel nitrous oxide tank due to a hard start with a paraffin hybrid. When the pyro (run) valve failed, a much larger volume of nitrous entered the preheated, flame-filled combustion chamber, and decomposed. Bits of combustion chamber went everywhere; we never found the injector."



The Pacific Rocket Society had a similar experience in the Black rock desert in 1995. A fill line leaked inside the combustion chamber, saturating the HTPB fuel with nitrous. Upon ignition the chamber blew up.

Warming the run-tank

On cold days, the **run-tank** has to be heated in order to get the nitrous pressure up (see our 'physics of nitrous oxide' paper). It goes without saying that care has to be taken to avoid hot-spots that could decompose the nitrous. If you're using an electrical tank heater, don't let it overheat.

Hot-spots within heat exchangers have been found to set off nitrous: use a heat exchanger coil with a uniform wall temperature such as a coil submerged in a water bath to warm nitrous.

Now that we know the hazards, what can be done about them?

The decomposition process

Know thine enemy: it's instructive to review the decomposition process. See Ref. 11 for a very detailed theoretical model, the results of which are summarised below:

Nitrous decomposition is a marginal reaction with just enough heat released to sustain itself. It is easily quenched: either nearby metal pipe walls or a diluent gas added to the vapour act to absorb heat and quench the reaction.

The decomposition rate of nitrous vapour is six orders of magnitude slower than peroxide decomposition, making it a safer oxidiser: it's less sensitive. This abnormally low reaction rate is partly caused by "the non-adiabatic spin-forbidden transition elementary unimolecular decomposition process" (it's a quantum thing: I don't understand a word of it either!)

All attempts in the literature to ignite liquid nitrous have failed (unlike liquid peroxide).

Small concentrations of diluent gas added to the nitrous vapour increase the ignition energy of the vapour, making the mixture extremely difficult to ignite at dilution levels greater than 30%. Adding extra gas to the **run-tank** is sometimes called 'supercharging', and has the side effects of raising the tank pressure slightly, and reducing the tendency for two-phase flow in the feed system.

Reference 11 defines two distinct modes of nitrous decomposition:

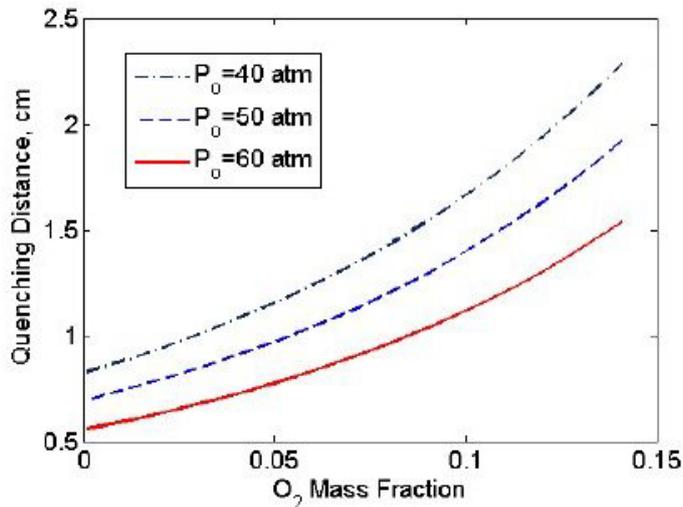
Local thermal ignition

This is regarded as the most common mode/ greatest hazard expected in nitrous rocketry, since it requires only small localised quantities of thermal energy. This starts a self-sustained deflagration wave in the nitrous vapour (starting at the injector, once all the liquid has run out) that travels up the tank, causing havoc (decomposing the rest of the nitrous vapour).

The rate of increase of tank pressure (the violence of the explosion) that this causes depends upon this wave's flame speed. The inherently slow decomposition of nitrous results in a low flame speed of only 15 cm/second at 41 Bar. The flame speed decreases with increasing pressure and increasing dilution of the vapour.

The quenching distance (the maximum radius of conductive metal pipe that will quench the reaction) is around 7 mm for pure nitrous at 27 degrees C (300 K), and increases with dilution with inert gasses:

the quenching distance estimate for N_2O/O_2 mixtures at 40, 50 and 60 atm for an initial temperature 300K. Note that the quenching distance for pure N_2O is around 0.7 cm and it increases with increasing level of dilution as expected.



Quenching distance for N_2O/O_2 mixtures at 40, 50 and 60 atm.

From a 'point' source or hotspot, the minimum ignition energy for pure nitrous vapour is very roughly 100 millijoules (not a lot). There is a big effect on local thermal ignition if you dilute the tank vapour: dilution by 30% Gox raises the ignition energy to roughly 500 Joules, making ignition virtually impossible.

[Note: as yet I'm not sure whether this dilution figure is based upon the mass of vapour at the end of the burn, or is based on the total nitrous mass at the start of the burn: I'm waiting to hear from the Space Propulsion Group.]

It may seem odd that adding Gox (gaseous oxygen) to the **run-tank** actually makes things safer. I assume it adds a bit to the **Specific impulse** as well.

Dilution with nitrogen is expected to have a similar effect, and helium is expected to be better: have the same effect at lower dilutions because heat gets conducted away more easily in helium. Ref. 10 suggests you need 4 times less helium compared to nitrogen.

The time taken for local thermal ignition-caused decomposition of the tank vapour to occur is surprisingly slow: a typical time to maximum overpressure is 5 seconds, (time to typical flight-weight tank burst is 2 seconds). This is slow enough for an automatic safety system to react and do something. Dilution doesn't really change the maximum overpressure.

Homogenous ignition

This is simultaneous ignition of a sizable bulk of the nitrous vapour when sufficient energy is added (high temperature, such as caused by a fire). It's regarded as the less likely of the two types.

Above a certain high temperature, the heat produced by the exothermic decomposition reaction exceeds the heat loss to the surroundings and the temperature rises.

A slow increase in temperature begins (an induction period).

This is followed by an exponential growth in temperature (causing very rapid gas expansion) once the temperature reaches the overheat temperature.

As this graph shows, below around 850 Kelvin, the induction period is too long for typical HPR propulsion systems (the nitrous has drained out of the tank and/or feed system by then) so auto-ignition is not going to happen.

Homogenous ignition is most likely to occur in the feed system due to reverse flow or adiabatic compression. It's unlikely to occur in the **run-tank** due to the need to heat the whole of the nitrous to its high 'auto-ignition' (short induction period) temperature.

The effect of diluting with inert gasses is small: even 80% dilution only raises the 'auto-ignition' (short induction period) temperature by 30 Kelvin.

Flame traps

There's been talk on Arocket of the possibility of adding a flame trap to the feed line. (Technically it's actually a quenching trap.)

Nitrous has a much larger quenching distance than typical fuel/air mixtures, so a grid of stainless steel tubes put streamwise into the feed line could be able to stop any decomposition that starts at the injector.

The quenching distance for nitrous is around 7 millimetres, so I reckon that a grid of 5 millimetre diameter tubes will work, and the tubes are large enough not to cause much of a pressure drop.

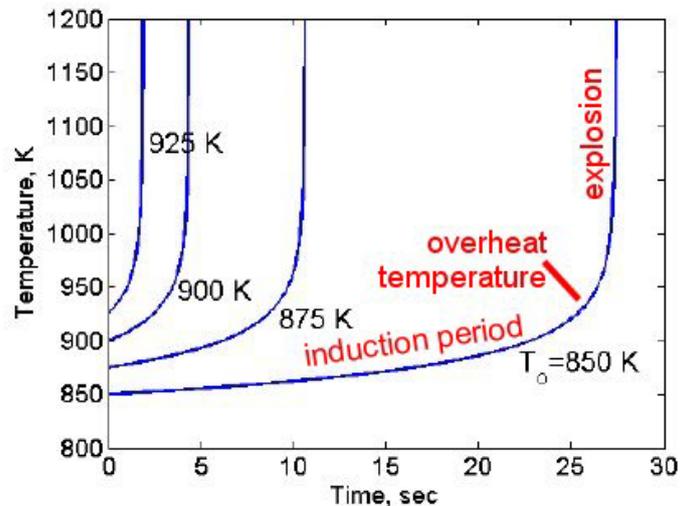
But I must stress that I haven't found any evidence of anyone having tried a flame trap with nitrous. This is a (potentially hazardous) experiment that needs doing.

Nitrous material compatibility

Use stainless steel!

As a general guide, materials that are oxygen compatible are suitable for nitrous.

It's worth pointing out that I haven't seen any evidence for a catalyst for nitrous that works at room temperature, they all seem to need elevated temperatures.



Induction time as a function of initial temperature, no heat loss case.



Metals

Aluminium, Stainless Steel: satisfactory

Copper and its oxides (and brass/bronze), nickel, and platinum, are highly catalytic with nitrous especially at elevated temperatures, so do not use these. Annoyingly this list includes the metals one would like to electroform into a thin-walled tank.

Carbon steels are corrosive in the presence of moisture: do not use corrosion-prone metals because for example, iron oxide (rust) is a catalyst. Avoid rust contamination from steel **fill-tanks**; use stainless steel filters in the fill line to catch any rust particles.

Plastics

Fluoroplastics such as Polytetrafluoroethylene (PTFE), Polychlorotrifluoroethylene (PCTFE), Fluorinated ethylene propylene (FEP), Perfluoroalkoxy polymer resin (PFA), are satisfactory. Other plastics are an ignition hazard.

Nitrous can saturate plastics and composites turning them into 'explosives': HTPB fuel saturated with nitrous can be 'explosive'.

Elastomers

Think carefully about what material to use as your run-tank O-rings. Just because commercial HPR hybrids use 'rubber' O-rings (nitrile rubber) doesn't automatically make it a good idea.

- Fluorocarbon-coated O-rings (Buzak and Shamban catalogue): use these where possible. Unfortunately, they don't stretch very much. Silicon (Q) **not silicone (see below)** is also satisfactory.

The main issue with other materials (the following list) is that they will slowly soak-up nitrous, leading to swelling of the material and an ignition hazard due to the absorbed nitrous. Limit nitrous exposure to a few hours at a time only:

- Buna-N (nitrile rubber NBR) and neoprene: will also degrade in nitrous liquid after several days.
- Butyl (isobutene - isoprene) rubber (IIR)
- Chloroprene (CR)
- Chloro-fluorocarbons (FKM) (VITON)
- Ethylene - Propylene (EPDM)
- **N.B. Silicone saturated with nitrous is an impact-sensitive explosive.**

Lubricants

Nitrous can form an explosive with many hydrocarbons and lubricants:

- Fluorocarbon-based lubricant (krytox): satisfactory
- Hydrocarbon-based lubricant: **definitely not recommended**, probable ignition hazard.

Recommendations

- Minimise the number of persons present in the test area whenever nitrous is being loaded/is loaded into the **run-tank** or whenever nitrous is flowing.
- Avoid decanting nitrous in small, confined areas (or monitor air quality) as nitrous is an anaesthetic.
- Use a large-volume (large diameter) **burst-disc** on the **run-tank**. Mandatory for manned systems.
- For engine static testing, keep the **run-tank** upright so only liquid gets to the injector at ignition.
- Keep it *very* clean: even very small traces of contaminating fuel can cause problems.
- Avoid the use of catalytic materials.
- Nitrous is a good solvent of hydrocarbons: grubby finger-marks, O-ring polymers, and valve seals.
- Venting the nitrous through the combustion chamber should be avoided at all costs.



- Dilution of the nitrous vapour in the **run-tank** is recommended.
- Avoid hard starts: fire the igniter before admitting the nitrous into the chamber.
- If using nitrous in a confined space use an oxygen sensor to monitor dangerous concentrations of nitrous which could be inhaled.
- Properly earth all ground support equipment (**fill-tanks** and fill lines), and keep mobile phones and laptop Wi-fi switched off when anywhere near nitrous. Persons handling nitrous should preferably be earthed.
- Construct tanks and lines out of conductive material, e.g. a metal internal tank liner. For composites, work out a way of dissipating static (aircraft composites have metal foil as their outer layer to dissipate lightning strikes. Another approach is to embed carbon nanofibres or nanotubes within the resin.)
- No part of the engine should have a resistance of more than one Ohm between it and its adjacent parts so that potential differences (voltage) cannot build up. (Ref. 1)
- Avoid adiabatic compression: minimise feed-line dead volume downstream of the run valve, slow the valve opening.
- Prevent back-flow of igniter gas through the injector.
- Reduce the pressurisation rate of your **run-tank** as you fill it to no more than 20 psi per second.
- Use small enough injector orifices and widen the nozzle throat to get a good pressure drop across the injector right through the burn (a drop of 20% of the combustion chamber pressure as the liquid runs out).
- Develop safety procedures - and pre-chill the pump - if you pump nitrous from the fill tank to the **run-tank**.
- Use **pressure relief valves/burst-discs** on all trapped volumes of liquid nitrous to prevent hydraulic overpressure.
- Review all moving parts in the nitrous system (regulators, valves) for friction, impact, and static discharge.
- Avoid eddies or stagnation zones in the feed pipework due to sudden changes in cross-section. These can act as flame-holders which prevent any decomposition from being flushed safely downstream.



Part 4: Liquid oxygen safety (Ref.s 1, 6, 17 – 21)

Lox Handling and safety issues

Liquid oxygen has a slight bluish tinge to its transparency. It pours like water though is slightly denser.

Oxygen is in fact one of the safer rocket oxidisers as it isn't corrosive, but in its cryogenic liquid form it is of course extremely cold, so spilling/splashing it on bare skin will cause severe 'cold-burns'. Note that Lox does not wet the skin for exactly the same reasons that liquid nitrogen doesn't: vapour pressure (the 'Leidenfrost effect' - a phenomenon in which a liquid in near contact with a mass significantly hotter than the liquid's boiling point - produces an insulating vapour layer keeping that liquid from boiling rapidly.)

Oxygen is a powerful oxidiser in both gaseous (Gox) and liquid (Lox) states and its use involves a degree of risk that you should never overlook.

Oxygen is reactive at ambient conditions, and its reactivity increases with increasing pressure, temperature, and concentration. Many materials that don't burn in air will burn emphatically in oxygen-enriched atmospheres and will do so with lower ignition energies and faster burn rates than in air. Most non-metals are flammable in 100 percent Gox at ambient pressure (or Lox), and most metals are flammable in Gox at increased pressure, or Lox.

Impressively destructive fires have occurred in every industry or venue in which oxygen-enriched atmospheres are used.

Typically, a flow of Lox from a **run-tank** through the rocket engine plumbing is preceded - and followed - by a flow of Gox, so review your plumbing for both Gox and Lox flow: both have their respective hazards.

Inhaling cold Gox at high concentration will severely damage your lungs: severe frostbite or actual ignition.

Infiltration

Gox - being a small diatomic molecule - has a habit of seeping into anything porous, such as foam insulation, clothing, or up trouser-legs. It can also get trapped in people's hair, and most hair cosmetics are utterly flammable. Avoid all hair 'products' and makeup/moisturiser on firing days.

Anyone coming in contact with a fair concentration of Gox (accidentally, or during filling of a **run-tank**) must stand outdoors - preferably in a breeze - for at least 30 minutes (Ref. 18) to let the Gox dilute and dissipate from their person and clothing, and also from their skin.

Waiting only five minutes for the Gox to dissipate then lighting a cigarette, or turning on the ignition of a car, or using a mobile 'phone or laptop Wi-fi, would all be extremely hazardous. And don't use any tools (when saturated with oxygen) which could cause a spark.

If oxygen escapes into an enclosed space or building, open all the doors and windows. Do not connect electrical power or carry out any maintenance for at least one hour (Ref. 18) after this event, to allow a potential oxygen-enriched atmosphere in the building to dissipate.

If using Lox in a confined space/indoors, use an oxygen sensor to monitor dangerous concentrations of Gox which could be inhaled.

Clothing

Protective clothing, goggles and gloves, are mandatory for those involved in any handling of Lox. BOC sell suitable gloves and aprons online. If there's any chance you might get sprayed with Lox or have to crawl through some in an emergency, dress accordingly.



Gloves for use around Lox systems must have good insulating quality. They must be designed for quick removal in case Lox gets inside them. If the gloves become heavily contaminated/soiled then they should be thrown away and replaced.

Because footwear may also get Lox inside, shoes must have high tops and trouser legs must be worn outside and over the shoe tops. The trousers must have no external pocket openings and no cuffs. The shoes should be made of leather.

When you're working with Lox, always wear natural fabrics (inside and out - no nylon underpants under your wool trousers) because plastic fabrics tend to melt and stick to the skin causing severe burns.

If you spill Lox on your clothes, get them off as quickly as you can. Several oxygen safety manuals - e.g. the one from NASA Lewis - say that clothing splashed with Lox or permeated with Gox can remain dangerously flammable for a surprisingly long time and should preferably be taken off at once.

Probably a better idea would be a safety shower. Hose down scrupulously, then take the clothes off while you are still under the shower. Clothing saturated with oxygen would turn you into a very good representation of a Roman candle.

Do not wear shorts around Lox!

Shoes for shop work that one can get out of very quickly if need be are recommended.

XCOR have the same rule for synthetic fabrics around Lox. It's not absolutely prohibited, but it's strongly discouraged: it can't be the only layer, and if it's an outer layer, you need to be able to take it off in two seconds. People get the hint pretty quickly, and generally only wear natural fabrics to work.

When working with oxygen, clothing must not be worn which produces sparks or static electricity, such as nylon, orlon, dacron, wool, silk, or shoes with steel taps or hob nails.

Warm water

Water hoses should be available to thaw valves and fittings on cryogenic storage containers. Atmospheric moisture may freeze on valve stems and similar components, making them impossible to open or close. Running water onto the frozen part may thaw the ice and enable component operation. Running water is also useful to thaw ice if someone's gloved hand freezes to a valve handle.

Explosive contamination

Liquid oxygen explosives use a cartridge of (typically) charcoal soaked in Lox. One charcoal brickette soaked in Lox has the explosive potential of a stick of 30% dynamite. (Needless to say, don't try this at home; it's friction, impact/shock, and static sensitive.)

Not as dangerous as a number of other materials, but a lot of stupid lab tricks with Lox and various organic materials can detonate in the hand rather than the floor they're thrown at... When it's well mixed with Lox, almost any organic material is an explosive - the details don't matter very much.

Just about anything that will burn may explode if sensitized with Lox: wood, paper, clothes, shoes. The Reaction Research Society had a Lox/tarpaper hybrid explode quite spectacularly ("detonate"), and it was attributed to an unsuccessful previous ignition attempt leaving the tarpaper saturated with Lox.

Dust, lint, or fine metal particles (swarf) can also cause an explosion, so don't do any metal filing, and only use lint-free cloths.



Lox and asphalt are a contact explosive. The rule is the obvious one, don't work with Lox on or over an asphalt surface. If you do spill Lox on asphalt, don't step in it! Apparently this happened at Edwards Airforce Base many years ago, and blew a guy's foot off.

The Lox evaporates, but the asphalt is still saturated with oxygen. It can remain explosive and impact-sensitive for hours at moderate temperatures.

At the very least, Lox and asphalt are highly flammable (asphalt has been used as a fuel in some hybrids): a Lox spill at Cranfield University U.K. in the 1960's burnt-out the staff car park and all the cars therein.

If you spill Lox on asphalt, cone it off. If there is a chance of anyone/traffic traversing the area for the next several hours, post a guard.

To reiterate: when working with cryogenic fluid

- Wear eye protection: goggles or face-shield.
- Wear the specified protective clothing: the gloves and apron.

When working with Liquid oxygen

- Hair and clothing will absorb gaseous oxygen while working with Lox.
- It will take at least 30 minutes for this gaseous oxygen to dissipate once you've left the test site.
- Avoid potential ignition sources (naked flames, lighters, soldering irons, cars, electrical equipment) while handling Lox, and for at least 30 minutes afterwards.
- Avoid wearing flammable cosmetics. (e.g. hairspray, brill-cream, hair-gel)
- Wear only natural fibres, and not ones that create static.
- Remove oxygen-saturated clothing.
- Ban all smoking.
- Take care with mobile (cell) 'phones: switch off all 'phones at or near the test/filling site as they have the electromagnetic strength to cause sparks. Switch off laptop Wi-fi's for the same reason.
- If oxygen escapes into a confined space, evacuate the building for one hour.
- Be aware that Lox mixed with organics can form an explosive.
- Do not use Lox over asphalt.
- Keep hands and tools free of dirt and grease: use degreased tools specifically nominated for use with the Lox plumbing only.
- Avoid the creation of dust, lint, or swarf around oxygen.

Team safety

Firing team member safety should be the paramount design driver for the rocket engine and its plumbing, and this is achieved mainly through remote actuation of the engine and its valves from a considerable distance.

Emergency operation

Be sure to hold several design reviews of the rocket engine's plumbing in order to make sure it can cope with all potential emergency situations. Try to design-in fault tolerance.

There must be facility to quickly shut-down the engine and dump the contents of the Lox **run-tank** well away from the test-stand. The outflows from both this drain/emergency dump valve, and the **run-tank burst-disc**, should flow down a pre-purged pipe of considerable length to dump the Gox well away from the engine, into a pre-prepared pit where the Gox can dilute with the nitrogen in the air and dissipate. (Ref. 17)

Friction, impact, and static discharge

Review all moving parts in the Lox system (regulators, valves) for friction, impact, and static discharge, which could ignite the oxygen.



Dewars

Lox is supplied in a double-walled insulated metal tank similar in design to a vacuum flask, which is known as a Dewar tank.

One person will have to manually connect and disconnect the Lox fill line filling the **run-tank** from the Dewar, and one or more persons will then have to move the Dewar well away from the test firing stand. Choreograph these person's actions and movements: ensure that they can carry out their allotted tasks in a safe manner.

Do not permit dirt, oil or grease to come in contact with the filling adapter on the oxygen cylinder or Dewar. Oil or grease, even in minute quantities, may cause a violent explosion upon contact with oxygen.

Do not drop the Dewar or it'll crack and spill Gox: use suitable trolleys/forklifts. Do not roll the Dewar on its edge.

Liquefaction

The air around uninsulated cryogenically cold metal pipes and tanks can become liquefied, separating into predominantly liquid nitrogen and liquid oxygen. This new Lox is a fire hazard. Insulate the tanks and pipework to prevent this. Liquefied air dripping onto asphalt or dirt/earth can ignite or form a contact explosive.

Oxygen fires

Oxygen fires cannot be put out.

Smothering the fire doesn't work, as the oxidiser remains in-situ. Try not to be the object on fire; they won't even recover your teeth.

You just have to let the fire burn out, though dilution of the oxygen may help.

In a Lox fire, the oxygen is typically boiling, and expanding by a factor of about 1000. This tends to blow the flames in every direction, whilst the oxidiser fans any secondary fires up well.

Once an oxygen fire has started in the rocket engine plumbing, the metal plumbing can then catch fire and be completely consumed, leading to further fire and a major oxygen spill.

Sources of ignition (Ref.s 1, 18)

The following is a list of some potential ignition mechanisms for oxygen:

- Particle impact: minute particles of metal becoming entrained in a high-speed flow of Gox and then impacting some component of the plumbing. The energy of impact is sufficient to ignite certain metals such as aluminium and titanium. Limit flow velocities, and use filters to catch any stray particles. Items that can generate particulate contamination during operation include pumps, check valves, rotating stem valves, regulators, and quick disconnect fittings.
- Rapid pressurisation (adiabatic compression: see later).
- Flow friction: The heat generated when oxygen at elevated pressures flows across or impinges on a polymer and produces erosion, friction, or vibration. Though poorly understood, this phenomenon can occur when maintenance or assembly damages the soft plastic of valve seats, leading to a leakage flow of oxygen across the seats. Using redundant seals can mitigate against this leakage.
- Mechanical impact: reactive metals such as aluminium, magnesium, titanium, and lithium-based alloys as well as some lead-containing solders can be ignited by mechanical impact in the presence of oxygen, as can many non-metals. Regulators, relief valves, and check valves that chatter (oscillate rapidly exposing their polymer components to repeated impacts) can ignite the polymers. Dropping a metal tool on asphalt saturated with oxygen can cause a minor explosion.
- Friction: two or more metal components rubbing together through rotational or translational sliding contact, generates enough heat to ignite.



- Resonance: acoustic oscillations within resonant cavities causes a rapid temperature rise. A flow of high-speed Gox through a tee-piece of plumbing which has one leg of the tee closed can form a buzzing cavity. Keep the plumbing internally aerodynamically smooth to avoid areas of turbulence.
- Static electricity discharge.
- Lightning: provide lightning rods, aerial cable, and suitably connected ground rods in all areas.
- Electrical arc: can occur from electric motor brushes, electrical power supplies, lighting, etc.
- External heat.
- Shockwaves and fragments from a ruptured tank.

Oxygen enrichment of the air due to a Gox spill greatly increases the hazards of flammable liquids, vapours and gases as follows:

- It widens the explosive limits. It does not affect the flash point significantly, so materials below the flash point remain safe. The increase is into the fuel rich area.
- It greatly reduces the energy required to produce ignition.
- It increases the rate of burning, and explosion relief becomes more difficult.
- Flame traps, flame-proofing of electrical equipment, etc. may become ineffective.

Another danger in the event of an upset is the mist explosion or torch effect when a mist or fine droplet spray of an organic liquid is discharged into an oxygen-enriched atmosphere. Under these conditions an explosion can occur regardless of whether the organic liquid is above or below its flash point.

Oxygen enrichment can also render flammable some gases and vapours which are not flammable in air, for example ammonia and some halogenated organics.

Oxygen as it affects test-stand design (Ref. 17)

Note: for Lox test-site design, see the final chapter of reference 18.

Gox is heavier than air and pools on the ground, so a sloping test-chamber floor with guttering will encourage the Lox or Gox to flow away from the test-stand, in this case into a prepared pit to let it pool, dissipate, or burn itself out. Don't let the oxygen enter drains, sewers, or streams. (You'll blow-up the sewer, and then...) Nitrogen in the air will eventually dilute the Gox as it dissipates.

The outflow from the dump/drain valve - especially during an emergency dump - should flow down a pre-purged pipe of considerable length, and then out into a pre-prepared pit. The outflow pipe downstream of the **burst-disc** should also be connected to this long dump line. This keeps the Gox well away from the test area.

If the wind direction would blow the Gox back again, no testing should be done.

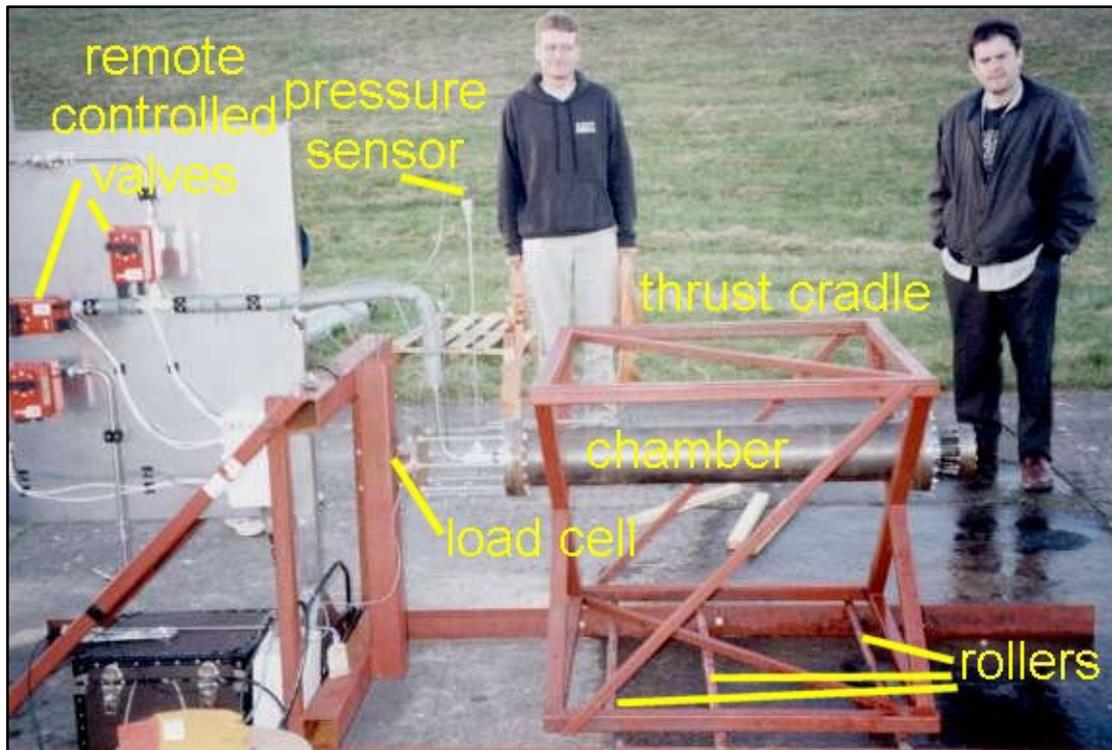
The walls and floor of the test stand should be concrete, (not earth or asphalt) and should be scrubbed using water only before every firing session to remove organic and/or particulate material (dirt) which could be a fire hazard.

The brooms or brushes used for this scrubbing should be bought new at the start of the testing programme, and be clearly labelled so that they won't accidentally be used elsewhere with detergents (which are flammable).

Any cracks in the concrete tend to act as dirt traps, and should be filled-in.

The test-stand and all equipment must be earthed.

Here's Aspirespace's test stand for our H₂O liquid oxygen hybrid:



The rollers let the rocket move a little to butt up against the load cell, which is an electrical device that converts force into an electrical signal. This measures the thrust, and sends it down an electrical cable to be read by a computer.

Not shown in the picture above are heavy straps that hold the chamber and thrust cradle down to the ground, but let it move along the rollers a bit.

Plumbing design

Cryogenically-rated hardware is worth the expense, because you save all the hassle of repeating 200 year's worth of cryogenic mistakes yourself. Not using cryo-rated hardware with Lox is like not using peroxide-compatible materials with peroxide. You'll probably get away with it, and it'll probably work most of the time. But it's not worth it, because the extra expense (damage/fire/explosion) when it *doesn't* work is higher than the savings when it does.

We recommend stainless steel pipework with Swagelock fittings. This allows easy and repeated disassembly for maintenance and modification.

Ball valves

Lox ball valves have to be vented; vented ball valves are a commercial off-the-shelf item. They're more expensive, but they're worth it.

The vent is there to release trapped Lox and/or trapped water-vapour: liquid water is the enemy in cryogenic valves, because it will freeze and the valve will stick. Trapped Lox will evaporate and build up pressurized Gox in the valve, where it will happily wait for an opportunity to ruin your day.

So if you intend to close a standard ball valve full of Lox, drill and de-burr a hole from the high pressure (inlet) side to the centre of the ball, otherwise expanding trapped Lox within the ball cavity may blow out the valve seal. This may not be necessary for the main run valve since it gets opened until all the Lox is gone.



The ball valve vent should have a short tube attached to it so that condensation won't occur on the ball or seal. Water at Lox temperatures is harder than steel.

Avoid rotating valve stems and sealing configurations that require rotation on assembly. Rotating valve stems and seals can gall and generate particulates.

Poppet valves

High pressures and high flow rates can produce side loads and oscillations on the poppet seal; these can cause metal deterioration by fretting or galling. (Galling is the more severe condition, because it involves smearing and material transfer from one surface to another.)

Fretting and galling can cause several problems in oxygen systems. The valve poppet may seize, resulting in loss of function. The frictional heat of the fretting or galling may lead to ignition of the valve. The particles generated by the fretting or galling may cause malfunction or ignition of another component downstream.

Where possible, the valve poppet should be designed for symmetrical flow so no oscillatory side loads are created. The symmetrical flow centres the poppet in the bore and maintains design clearances between the poppet and bore surfaces.

Check valves

Bear in mind that check valves (one-way valves) have a minimum back-pressure below which they will either not actuate, or will 'chatter'. Check the information that comes with the valve.

You should have a check valve on the **run-tank** vent. For example, use a 1.5 psi cracking pressure ball check valve in 'Teflon' and stainless steel on the outlet (XCOR). This keeps the system nice and dry with the vent wide open, and means that the tank contains only oxygen (and possibly helium) after the first fill cycle. This is good for keeping everything dry and clean.

Welded or soldered joints

Welded, brazed, or silver-soldered joints are satisfactory for oxygen systems. However, such joints - if left in the as-formed condition - may have slag or surfaces that can trap contaminants.

Welds should be specified as 'full penetration' so that the contracting surfaces are joined to limit particulate entrapment. Exposed weld surfaces should be ground to a smooth finish for ease of cleaning.

With brazed and soldered joints, special care must be taken to ensure surface cleanliness, close and uniform clearance, and full penetration of the joint.

Thermal expansion

Design for pipework thermal expansion and contraction with cryogenic fluids. Leaks are commonly caused by the disparity of thermal expansion coefficients between polymers and metals. Upon cooling, the shrinkage of polymers will exceed that of metals, and seals will lose the compression required for sealing.

Flexible hose

Don't let flexible hose flap about: include a maximum allowable slack of about 5 percent of the total length, and use restraining cables and anchors for the hose. Consider using a spring of coiled stainless steel tubing rather than hose, as hose is more flammable.

Maintenance

All equipment in any oxygen system that may be removed for inspection, maintenance, replacement, etc., should be provided with a vent valve for blowdown and purging, and to make sure it really is depressurised before removal (we got that wrong one time - a nasty bang - but no harm done).



Blunt flow impingement surfaces

Minimize internal blunt flow impingement surfaces. The risk of particle impact ignitions can be reduced if potential impact surfaces are designed with shallow impact angles to reduce the kinetic energy absorbed by the impact surface upon impact.

Burrs and sharp edges

Eliminate internal burrs and avoid sharp edges. Burrs and sharp edges on equipment provide ignition sources for particle impact, and they also provide the ingredients for a 'kindling chain' (the promotion of ignition from materials of low ignition temperatures to materials of higher ignition temperatures) combustion propagation. Removal of this material is standard shop practice and is essential to avoid oxygen-enriched ignitions.

O-rings

Installation of an O-ring over threads with an outer diameter exceeding the inside diameter of the O-ring should be avoided if possible or the O-ring gets nicked/cut.

Press Fits

Press fits generate particulate during their assembly from the relative motion of the two highly loaded surfaces.

- The particulate can be partially removed by cleaning the joined parts immediately after pressing them together.
- Assemble press-fit, push-fit, and threaded valve parts into housing bores with the housing inverted (bore opening pointing down), so that contaminants generated during assembly fall away from the component rather than into flow paths.
- Cleaning components with press-fit parts is extremely difficult.
- These parts should never be submerged into a cleaning solution or bath, as the cleaning solution enters between the two press-fit parts, leaches out later, and becomes a contaminant.

Blind passages

Eliminate blind passages. Long, narrow internal passages or blind passages are difficult to clean and to inspect for cleanliness. Additionally, they can provide a location for particulate to accumulate during operation of the equipment. This contamination can make the equipment susceptible to particle impact, rapid compression, and resonant cavity ignitions.

Crevices and cavities

Avoid internal crevices for particulate entrapment and resonant cavities. Cavities - especially those formed at the intersection of mating parts in assemblies - create a location where contamination can accumulate and increase ignition risks, as in blind passages.

Filters

Use filters to isolate system particulate; however, they should be placed in locations where they can be removed and inspected and where no possibility of back-flow exists.

Use filters at the following locations:

- Module inlets and outlets.
- Disconnect points.
- Points required to isolate difficult-to-clean passageways.
- Upstream of valve seats.

Choked flow

Design to minimize choked flow (transonic or supersonic flow velocities). If high flow speeds are probable, consider shut-off valves, metering valves, **pressure relief valves**, and regulators, to reduce flow speed to reduce the kinetic energy of particles (impact ignition risks).

Captured vent systems

Opinions differ about the merits of captured vent systems: some say they should be avoided, but they do prevent dust and moisture coming into the system (see 'ball valves' above).



A **pressure relief valve** or **burst-disc** that is not open directly to the atmosphere - but rather has a tube or pipe connected to the outlet - is said to have a captured vent.

If a captured vent is necessary, use highly ignition-resistant materials such as Monel or copper, and make sure the vent outlet empties well away from personnel (use shields or barriers as appropriate) and vents to outdoors. Consider a screen on the outlet to prevent insects and/or small mammals crawling into the pipework.

Flame-holders

Avoid eddies or stagnation zones in the feed pipework due to sudden changes in pipe cross-section. These can act as flame-holders which prevent any fire from being flushed safely downstream.

Seals

Soft materials for seals are often necessary. But they can cold-flow (permanently deform) with use. Also, soft seals can extrude under high temperature and pressure, generating particulate contamination.

High-temperature seals (e.g. for the combustion chamber) are often copper, however the resulting copper oxide can dislodge and generate particulates.

Vaporisation

Be aware that Lox - if accidentally allowed to warm up - will convert to potentially very high pressure gas. Design the plumbing and operating procedures to avoid this, and mount safety devices (**burst-discs** and **pressure relief valves**) where Lox could become trapped.

Letting the Lox drop in pressure - due to a decrease in plumbing diameter and Bernoulli's principle - encourages the Lox to vaporise, which can cause downstream adiabatic compression problems, and 'geysering' of the Lox (major bubble formation). Try to avoid this by keeping the plumbing diameter constant and wide (resulting in a Lox flow velocity less than 10 metres per second).

Bowing

Horizontal pipelines may experience cryogenic bowing because of stratified flow (warm Gox bubbles on top, cold Lox below) or because a single liquid Lox layer exists on only the bottom of the pipe. The large forces normally generated by bowing should be considered when designing pipe-guide supports.

Leak testing

After the plumbing has been **hydrotested**, you may want to test for leaks using leak test compound. This is a sort of washing-up liquid that you coat joints with and look for bubbles forming.

Oxygen systems leak test compound must be used for leak testing oxygen systems.

The leak test compound must be inspected before applying it to oxygen system components. Leak test compound must be clear, free from suspended material and/or sediment, and peculiar odours such as acetone or alcohol. Leak test compound which does not meet these qualifications must be disposed of.

Leak test compound is highly corrosive and must be removed from oxygen system components upon completion of the leak test.

Heat leakage

A little heat from the environment leaks into even the best insulated of tanks, especially if the Lox has to stand in the Lox tank for any period of time. Some of the Lox will always gasify.

Lox has a high vapour-pressure compared to most liquid oxidisers (excepting nitrous oxide), requiring thicker walled, heavier tanks; but it's still very small.



For hybrid **run-tanks** externally-pressurised by inert gas, the Lox vapour pressure isn't an issue, as the inert gas pressurant is at a much greater pressure, and this sizes the required tank strength.

Should the pressure exceed allowable limits, this is simply handled by having a valve known as a **pressure relief valve** that will open at a set pressure to bleed off the Gox, and for added safety, incorporate a **burst-disc** in parallel that will break and vent the tank to the atmosphere if the Gox reaches a set overpressure.

Each section of Lox piping capable of being physically isolated should be considered a pressure vessel in its own right with a source of heat into the pipe. Add **pressure relief valves** accordingly.

Pipework should be insulated; long pipes need vacuum jacket insulation.

Cryogenic cold-shock

Cold-shocking a newly-assembled Lox system by loading it with clean liquid nitrogen following final assembly is highly recommended.

After the cryogenic cold-shock, the system should be emptied of liquid nitrogen and warmed to ambient temperature. Bolts and threaded connection must then be retorqued to prescribed values, and gas leak-checking procedures should follow.

Following cold-shock, the entire system should be inspected for evidence of cracking, distortion, or any other anomaly, with special attention directed to welds. Then system cleanliness must be checked and verified.

Lox tank construction

The **run-tank** should be designed with a safety factor of at least 2.0 for ground-handling/propellant loading safety as advised by missile-design literature. For our H2O Lox hybrid we are using a commercial aluminium diving cylinder rated even higher.

Several metals used for tank construction - such as aluminium alloys - actually get stronger at cryogenic temperatures, therefore it makes more sense to put the required thick foam insulation onto the *outside* of the metal wall.

An ensuing design problem is the fact that there must be a perfect bond between the tank wall and its insulation jacket. If any air-bubbles (known as voids) exist between the insulation and the tank, the cryogenic temperatures of the metal wall of the tank will cool these air bubbles enough to liquefy them, causing puddling which peels the insulation.

To what extent the air bubbles liquefy depends upon the wall temperature, i.e. what's in the tank: cryogenic nitrogen would tend to produce puddles of mostly liquid nitrogen, whereas Lox tends to produce Lox puddles, which is a serious fire hazard.

The foam insulation on the Lox **run-tank** must therefore be fireproof, carefully bonded, and preferably of low porosity.

Wiring

Electrical wiring inside Lox tanks should be encased in hermetically sealed conduits or conduit inerted with helium or nitrogen gas. The instruments, switches, flow sensors, and electrical devices should be designed in modular structures and hermetically sealed, and inerting with nitrogen or helium is recommended.

Shutoff valve

An emergency isolation valve that stops Lox flow from the **run-tank** in case of a plumbing failure downstream should be provided as close to the tank as possible.



Purging and cleaning

For safety, every last speck of air and/or water (vapour) has to be removed from the Lox **run-tank** and all the Lox plumbing before filling with Lox can begin, because any remaining moisture could freeze a valve in the plumbing stuck, for example, causing a runaway engine and possibly an explosion.

The last Ausroc flight suffered from a frozen valve, and so we were advised to slowly move several of the valves a tiny amount at regular intervals to keep them unstuck. Though some Lox/Gox will then escape, this should help with the chill-down. (See below.)

At the same time, everything must be clean and free from oil or grease ('Krytox' oxygen-compatible grease is okay). Every speck of dust or weld/braze solder flux, or finger-marks (anything organic) have to be removed from the Lox tank and Lox plumbing as well, because many organics spontaneously ignite when they come into contact with Lox.

This would create a mini-fire, and because the ignition temperature of most metals with Lox is very low, this fire would then ignite the metal of the Lox pipework.

The worst scenario is if the fire reaches - or starts within - the Lox **run-tank**, causing an excessive build-up of pressure within the tank that might blow the tank apart despite the **burst-disc**.

The traditional solution to all of the above problems is known as 'purging', wherein nitrogen gas (which is chemically inert) is forced into the tank and plumbing for sufficient time to expel all the impurities (air, water-vapour and dust) just prior to the Lox filling operation.

Do not use a mechanical compressor because compressed air always has some oil in it from the air compressor. Use UPC grade nitrogen gas from a cylinder.

Everything must be dry; ball valves should be dried in both open and half-open positions to dry out behind the ball.

Put a vacuum cleaner on one end of the system and a hot air gun on the other end. Put a filter on the intake of the hot air gun: there's plenty of potentially combustible dust in your average workshop.

More stubborn organic impurities are dealt with by scrupulous cleaning of all re-used components in special fluids such as **trike** or soaking the parts in acetone or Isopropyl alcohol for a while before the soap and de-ionised water wash.

Cleaning is much more effective if the plumbing is broken down into component parts that are individually cleaned.

Wear clean gloves during and after cleaning.

Cleaning individual components – a typical commercial procedure

- Dust and loose contaminants are removed by scrubbing, then rinsed in water.
- Ultrasonically clean the part.
- Clean with oxygen cleaning agent.
- Alkaline clean, then acid clean, or use detergent for aluminium and non-metals. Alkaline is okay for Teflon, acid is not okay for welded stainless steel joints: use acid pickling bath (3-5% hydrofluoric acid and 15-20% nitric acid for 5 minutes to remove welding discolouration and slag). Caution, nitric acid pickling of copper or brass can result in nitrogen dioxide emissions. Local exhaust ventilation must be used as necessary to prevent exposure of personnel to this highly toxic gas.
- Rinse and soak in de-ionised water three times to remove the oxygen cleaning agent.
- Dry the parts with nitrogen.
- Inspect cleaned parts, re-clean if they fail.



- Visual inspection looking for loose contaminants or grease.
- Wipe with a white lint-free cloth (check that the cloth remains clean).
- Inspection using an ultraviolet lamp will reveal fingermarks/smears.

Store all cleaned parts in sealed plastic bags and re-inspect just prior to assembly.

Assembly/reassembly cleaning cycle (typical)

After cleaning each item separately as above and re-assembling the plumbing in a clean room environment (gloves and coveralls):

- Flush system with water and a grease removing agent. Cycle ball valves to clean out the area behind the ball i.e. run for 10 minutes half-open, then 10 minutes wide open etc.
- Flush system with distilled water.
- Dry the system. Suck air through the system with a vacuum cleaner and put a heat gun on the system inlet (not too hot, or fluoroplastic valve seals could melt). Run until the whole system is hot and dry, Cycle ball valves as above. Leave the system shut until ready for use. Do not have cryogenic ball valves exposed to the air without a foot-long tube downstream of the valve to prevent condensation on the ball.
- Blow/purge entire system out with compressed nitrogen.

Bagging

For the first assembly of the engine, manufacture the components in a 'clean-room' and bag-seal all assembled components until firing, when, for example, the rocket exhaust burns through a thin plastic film sealing the nozzle. Special non-residue (clean room) adhesive tape can be bought for such applications.

For larger lengths of pipework, plug the ends of the pipe with plastic bags or plastic plugs, do not use cloths or rags.

Cleaning ethos

Experience shows it's better to start with a very clean system and keep it that way, rather than try to clean a contaminated system. (Ref. 18)

Proper record keeping will tell you what's been cleaned and when.

After each component has been cleaned, wear gloves to prevent natural skin oils from contaminating the component from then on.

Remember that regulator contaminants could get carried backwards into the Lox tank, so the full cleaning is advisable. If you don't do that, at least put a good filter between the regulator and the Lox tank.

The filling operation

Filling the Lox **run-tank** is a straightforward procedure, though as much of the operation as possible should be done by remote-control for safety.

One problem is that the thermal shock of pouring cryogenic Lox onto the tank walls (and also the plumbing when the engine starts) could fracture the metal or 'craze' a composite tank.

Even if the thermal strains weren't sufficient to cause fracture, they still subject the tank and its mountings/ plumbing to severe distorting forces.

The thermal shock can also lead to 'geysering' (imagine Lox spouting like a shaken fizzy drink).

Purging the system with very cold gas for sufficient time to pre-cool the plumbing helps considerably. This is known as a 'chill-down'.



With a fresh tank at 230 bar, the Aspirespace H2O purge system uses nitrogen which has isentropically cooled down to around 90 Kelvin during the expansion to around one atmosphere. Controlled release of small amounts of Lox/Gox will also help the chill-down.

Filling of the Saturn 5 tanks was preceded by a controlled chill-down of the system, by purging with increasingly colder helium to reduce thermal shock when the Lox was subsequently poured in. The Lox was poured in at a precisely calculated rate: slowly at first to reduce the amount of 'coke-bottling' (where the tank shrinks in diameter at the point where the Lox first touches the walls, to resemble the old-style small glass coke bottle).

The Aspirespace H2O Lox **run-tank** is filled from a Dewar tank:

- The vent valve on the Dewar is briefly closed, allowing it to self-pressurise, in order to pump the Lox down the filling hose to our **run-tank**.
- We fill our **run-tank** from the bottom-up, keeping our vent-valve open to let the displaced nitrogen from the purge flow out as the Lox flows in.
- We can tell when our **run-tank** is full, as Lox will pour out of the vent line. (By arranging our vent pipe to go almost - but not quite - to the top of the tank, this leaves the necessary space for Gox boil-off.)

The filling operation should continue until just a minute or so before firing, because some heat from the environment will seep through the **run-tank** insulation, causing some of the Lox to boil into gas which ends up flowing out the vent valve. This lost Lox has, therefore, to be continuously replaced (topped-up) until just before firing.

Adiabatic self-compression

This phenomenon was discussed earlier in relation to nitrous oxide, and occurs mainly due to bad plumbing design and bad operation.

If a valve suddenly opens and Gox at high pressure shoots down a pipe that was initially at a much lower pressure, the Gox flow speed can go sonic (near-sonic is bad enough) especially if the pipe is too narrow.

Obstructions such as a closed valve, a sharp bend in the pipe, a burr protruding into the flow, etc. should be avoided, as the Gox will decelerate rapidly if it encounters these.

Molecules of gas at low pressure (i.e. low density) have enough inertia compared to their separation from each other that if the gas hits an obstruction as just described, the gas will 'pile up', that is, briefly self-compress, and this compression wave will bounce up and down the pipe a few times until it damps out.

The temperature of a gas rises if it's compressed, and if the gas was moving at high velocity prior to hitting the obstruction, this self-compression happens so rapidly that it's practically adiabatic, ergo there physically isn't time for heat to flow out of the gas into the surroundings, so the temperature can rise significantly.

The problem with Gox is that this high temperature (with a pressure differential of 40 bar, this could be an increase of nearly 500 degrees C) may well be above the auto-ignition temperature of non-metal components within the plumbing such as a valve seats, especially as the ignition temperature of most materials in Gox is lower than that in air.

Even if the plumbing doesn't ignite, the auto-ignition temperature of any grease or other organics in the system will be exceeded, and the ensuing local fire then sets the pipe material alight.

Basically, causing compression pulses or waves in any part of the system containing low pressure Gox should be avoided at all costs.



For a Lox hybrid, these dangerous conditions could occur during engine start-up because of Gox in the injector manifold, and any Gox further upstream caused by heat leaking through the pipe insulation into the Lox next to the walls of the pipe.

A severe pressurisation failure could also lower the system pressure to low values that possibly could suffer an adiabatic self-compression if a shut-off valve was slammed shut.

This is why all valves in the Lox system must be opened and closed slowly, therefore pyrotechnically operated valves or explosive pyrotechnic ignition systems must not be used. (Pyrovalves are okay in the nitrogen pressurisation system though.)

Suddenly opening the run-valve that allows the Lox to reach the engine could cause adiabatic compression if the Gox ahead of the Lox hits the injector at high speed.

Lox hybrid firing

Use small enough injector orifices and widen the nozzle throat to get a good pressure drop across the injector right through the burn (a drop of 20% of the combustion chamber pressure).

Use **pressure relief valves/burst-discs** on all trapped volumes of Lox (e.g. trapped between closed valves) to prevent hydraulic overpressure.

Prevent back-flow of igniter gas through the injector.

Lox hybrids have the reputation of running rough. The reason is that cold droplets of Lox break through the flame zone and boundary layer within the fuel port and impact the fuel grain surface, where they cause tiny explosions.

The way to cure this is to pre-heat the Lox to gasify it before it reaches the fuel grain.

There are a number of ways to do this; Aspirespace are planning to regeneratively cool the nozzle with supercritical oxygen (which must be at at least twice oxygen critical pressure to avoid flow irregularities) so as to heat the oxygen before it enters the injector. Another option is to use a heat exchanger or even another mini-hybrid within the combustion chamber just ahead of the fuel-grain.

Non-compatible flow-meters

A turbine-type flow-meter gives an accurate and direct reading of the flowrate down the pipe between Lox **run-tank** and injector, but incompatible turbine flow-meters in Lox are dangerous.

What can happen is that the relatively high temperature of the turbine rotor can locally vaporise the Lox, and the ensuing rapid expansion of gas spins the turbine (just like in a jet engine) up to phenomenal speed, whereupon it breaks up and the 'hot' bits of broken turbine get carried downstream in the flow and cause mayhem, by blocking the injector manifold or injectors for example.

Several liquid engines using Lox have blown up, because there were no debris filters in the system, and the bits of dead flow-meter got eaten by the Lox turbopump, which then exploded.

Lox-compatible turbine flow-meters do exist, but are unsurprisingly exceedingly expensive.

Non-turbine Coriolis-effect flow-meters have no moving parts and are therefore safer and slightly cheaper.



Materials compatibility: metals

The fire hazards inherent in oxygen systems make materials selection a crucial step in designing and maintaining a safe system.

You must have an understanding of the numerous factors relating to the selection of suitable materials for oxygen service: such as the material properties related to the design and operating conditions, ignition and combustion behaviour, property changes that occur at cryogenic temperatures, and ease of cleaning.

Most oxygen systems are metal, because generally, metals aren't easily ignited (though metal particles ignite easily). When they *do* ignite however, metals cause more damage than burning non-metals because of their higher flame temperature, and because they produce liquid combustion products that spread fires easily. You've got to see a metal plumbing fire to believe it! (The entire plumbing is consumed.)

Note that metals - including those that normally exhibit a high resistance to ignition - are more flammable in oxygen when they have thin cross sections such as thin-walled tubing, or when they are finely divided such as wire mesh or sintered filters.

Beware of welding compatible metals with non-oxygen-compatible welding rods.

The extent of the low-temperature embrittlement problem is much exaggerated. It doesn't really afflict all *that* many metals. The only reason it gets so much press is that our most common structural metal - ordinary carbon steel - is one of the ones greatly affected, so must be avoided.

If you're working with Lox, this is something to beware of. There have been cases where a minor Lox spill became a major accident because it caused brittle failure in steel components and the problems cascaded.

Most suitable

A number of metals are particularly oxidation resistant, either for chemical reasons, or good oxide protection. Most resistant are copper, bronze, nickel, many nickel alloys (e.g. Monel), and gold.

Nickel and nickel alloys are very resistant to ignition and combustion, and have significant low-temperature toughness.

Monel alloys, (nickel-copper alloys), are the least ignitable alloys commonly used as structural materials, and have a higher-strength-to-weight ratio than aluminium. [Note: Monel and Monel alloys are flammable in finely divided configurations, such as wire mesh and sintered powder.]

Inconel 718 (nickel-iron alloy) has been used extensively in oxygen systems, though isn't much better than stainless steel. Inconel MA 754 is superior to 718 and doesn't support self-sustained combustion.

Some Hastelloy such as C-22 and C-276 are much more ignition resistant than stainless steels or Inconel 718.

The 9% nickel steels are okay for Lox or liquid nitrogen (though don't let them rust). They are approved down to liquid nitrogen temperatures and are what most large commercial facilities use. At liquid nitrogen temperatures it's well on its way to becoming brittle, it just hasn't quite got there yet.

Copper and copper alloys are very suitable for Lox and Gox systems. Particularly useful for resisting ignition by particle impact and therefore can be used as impingement plates.

Note that copper oxide is fragile: tends to slough off and contaminate the plumbing.



Aluminum-bronze alloy - although containing a high amount of copper - is not recommended for use in Lox systems because of its flammability and ignitability.

Sintered bronze is less flammable than sintered Monel 400 and stainless steel for filter element material.

Stainless steel has been used extensively in Lox systems but is known to be more flammable and more easily ignited than nickel and copper-based alloys. Stainless steels are far more ignition and burn-resistant than titanium and aluminium alloys.

- The 300-series austenitic stainless steels are generally compatible with Lox.
- The classic Atlas rocket was made out of 301, as was Centaur.
- Beware that excessive cold-working can cause austenite-to-martensite solid phase transition in 301, after which it's brittle at low temperatures; this also limits fatigue life.
- 310 doesn't do that and would be preferable for long-life applications.
- 304 is good for cryogenic applications.
- 301 is approved, but not 301N. (301 is used extensively for Lox, whilst 301N goes partly martensitic at low temperatures and is not considered safe for cryogenic use.)
- Incidentally, 301 has excellent deformation characteristics, which make it good for spinning or complex pipe work. Given the cold-working problem it may not be suitable unless annealed after working, but it's worth a look.
- The nitrogen-bearing stainless steels have about twice the annealed yield strength of the 300 series but are expensive.

Aluminium alloys are attractive candidate materials for pressure vessels and other applications where no credible ignition hazards exist because of their high strength-to-weight ratios. However, the use of aluminium alloys in lines, valves, and other dynamic components should be avoided whenever possible because of their poor ignition and combustion characteristics. Aluminium burns really well in pure oxygen!

- Aluminium is ignited exceptionally easily by friction because the wear destroys its protective oxide layer, so it shouldn't be used in systems where frictional heating is possible.
- Aluminium is very easily ignited by particle impact, and aluminium particulate is a far more effective ignition source than many other metal particulates. Systems that use large areas of aluminium alloys in storage tanks should be designed to ensure that aluminium particulates cannot cause ignition of other metallic materials downstream from the aluminium.
- Promoted combustion tests on aluminium-lithium alloys have indicated that they are less flammable than aluminium.
- Particle impact tests on anodized aluminium targets have indicated that anodizing the surface increases the resistance to ignition by particle impact.
- The Saturn V used 2219 and 2014 for its tanks.
- 5052, 5086, and 5456 were all used successfully in the earlier von Braun rockets, but are not terribly strong.
- Thor and Titan were mostly 2014, despite the difficulties of welding it.
- 2024 is good.
- The Space Shuttle external tank Lox and LH2 tanks were 2219 until the switch to 2915 aluminum-lithium.
- 6061 is okay.
- The 7000 series is generally not recommended for cryogenic service, although 7079 in particular is okay for Lox.

Titanium is very iffy for Lox, depends on the exact alloy and can be very sensitive to small amounts of contamination. Titanium is impact-sensitive in oxygen.

6Al-4V was used successfully for Atlas's helium bottles, which are liquid nitrogen-cooled until just before launch to increase the helium density. A110AT also seems good.



Titanium's chemical affinity for oxygen is great (like most metals) but is a hazard in that a titanium tank filled with Lox is uneventful until the metal oxide coating on the titanium is disrupted, such as during pressurization, scratching the surface, etc. Explosions have resulted.

There are titanium pressure vessels floating around in the surplus market and these could be mistaken for stainless steel i.e. non-magnetic. Amateurs beware. Density and/or a spark test will tell the difference: Titanium has a density of around 4500 kg/m³ versus 7480 to 8000 kg/m³ for stainless steel. The spark test yields brilliant white sparks with titanium and dull orange for stainless steel.

Tin is Lox compatible, but tin 'C' rings lack the compliancy of spring-energized face seals for sealing.

Iron oxide (rust) is not a protective coating for the iron in Lox plumbing, the oxygen gets right through it.

Cadmium, beryllium, magnesium and Mercury: These metals are unsuitable due to their flammability. Cadmium and beryllium produce toxic gasses. Mercury and its compounds can cause accelerated stress cracking of aluminium and titanium alloys. Toxicity further limits its use.

Surface preparations: -Try to avoid coatings, platings, or hard-facings as much as possible.

Materials compatibility: non-metals

Most non-metals ignite easily and are highly flammable in Lox therefore the use of these 'soft goods' should be limited as much as is physically possible.

Fluorinated plastics 'fluoroplastics' - such as Polytetrafluoroethylene (PTFE), Polychlorotrifluoroethylene (PCTFE), Fluorinated ethylene propylene (FEP), Perfluoroalkoxy polymer resin (PFA), - are satisfactory as they require a high temperature for autoignition, release relatively low amounts of heat when burned, and require high concentrations of oxygen to be flammable. They also are flexible at cryogenic temperatures due to their extremely low glass transition temperature.

Silicone rubber is often used for cryogenic oxygen - again due to its extremely low glass transition temperature - but it has poor ignition resistance and has generally been replaced with DuPont 'Kalrez' per-fluoro-elastomer. Other rubbers burn easily.

Nylon has been used in oxygen systems when its superior mechanical properties over fluoroplastics are needed, but it ignites and burns more easily.

Take care assembling plastic valve seats so that 'hairs' of plastic don't form. They ignite easily.

Some composite fibres are Lox-compatible, but their resins are not unless the resin is a fluoroplastic.

Lubricants should be restricted to fluoropolymer greases such as 'Krytox', 'Braycote', or 'Fluorolube'. Note that these lubricants can cause severe corrosion if moisture is present.

Concentrated Gox/Lox hazards to human health

- Oxygen toxicity is a condition resulting from the harmful effects of breathing molecular oxygen (O₂) at increased concentration. It is also known as 'oxygen toxicity syndrome', 'oxygen intoxication', and 'oxygen poisoning'.
- Severe cases can result in cell damage and death, with effects most often seen in the central nervous system, lungs, and eyes.



- The result of breathing increased concentration of oxygen is 'hyperoxia', an excess of oxygen in body tissues. The body is affected in different ways depending on the type of exposure.
- Central nervous system toxicity is caused by short exposure to high concentration of oxygen at greater than atmospheric pressure.
- Pulmonary and ocular toxicity result from longer exposure to increased oxygen levels at normal pressure. Symptoms may include disorientation, breathing problems, and vision changes such as myopia.
- Exposures to very high concentration can cause oxidative damage to cell membranes, collapse of the alveoli in the lungs, retinal detachment, and seizures.
- Oxygen toxicity is managed by reducing the exposure to increased oxygen levels.
- Studies show that - in the long term - a robust recovery from most types of oxygen toxicity is possible.

External exposure to Lox (spillage)

- Get every item of clothing off immediately.
- Get hosed-down with tepid water for many minutes.
- Get prompt medical attention for severe frostbite: you may feel no pain until frozen tissue thaws out, at which point you *really* want access to *serious* painkillers.

Death

Unfortunately, the consequences of a blast of high-concentration oxygen right in the face can be VERY BAD, so don't let this happen.

There's no point pussy-footing around; aviators are warned not to walk into rotating propellers because they will die.

If you get a blast of any type of cryogenic fluid right in the mush, CLOSE YOUR EYES AND DO NOT INHALE (resist the instinct to go "Waah!"). Cryogenic fluid will literally freeze your internal tissue. This may kill you. Alternatively, when the frozen tissues thaw-out, the searing pain will make you wish you *had* died.

This can actually get worse: if the fluid in your lungs is Lox, it may combust your lungs, which is beyond horrible.

Basically, your chances of survival absolutely depend upon you NEVER getting into these situations.

To sum up, here are a list of recommendations for oxygen sequences

- Minimise the number of persons present in the test area whenever Lox is being loaded/is loaded into the **run-tank** or whenever Lox is flowing.
- Wear only non-static natural-fibre clothing.
- Use a large-volume (large diameter) **burst-disc** on the **run-tank**. Mandatory for manned systems.
- Warm water on tap can unfreeze things.
- Keep it very clean: even very small traces of contamination can cause major problems.
- Avoid hard starts: fire the igniter before admitting the Lox into the chamber.
- If using Lox in a confined space use an oxygen sensor to monitor dangerous concentrations of Gox which could be inhaled.
- Properly earth all ground support equipment (fill tanks and fill lines), and keep mobile phones and laptop Wi-fi's switched off when anywhere near pure oxygen.
- Construct tanks and lines out of conductive material. For composites, work out a way of dissipating static (aircraft composites have metal foil as their outer layer to dissipate lightning strikes. Other approach is to embed carbon nanofibres or nanotubes within the resin.)



- Avoid adiabatic compression: minimise feed-line dead volume downstream of the run valve, slow the valve opening.
- Prevent back-flow of igniter gas through the injector.
- Use small enough injector orifices and widen the nozzle throat to get a good pressure drop across the injector right through the burn (a drop of 20% of the combustion chamber pressure).
- Use **pressure relief valves/burst-discs** on all trapped volumes of Lox to prevent hydraulic overpressure.
- Review all moving parts in the Lox system (regulators, valves) for potential particle impact, friction, mechanical impact, and static discharge.
- Avoid eddies or stagnation zones in the feed pipework due to sudden changes in cross-section. These can act as flame-holders which prevent any fire from being flushed safely downstream.
- Avoid resonant cavities, flexible hose, blunt flow impingement surfaces, burrs and sharp edges, blind passages, crevices, choked flow.
- Avoid non-compatible turbine flow-meters.
- Carefully consider materials compatibility in the system.
- Minimise the use of non-metals.
- Use only approved leak test compound.
- Cryogenically cold-shock new plumbing.
- The Lox fill tank should have an emergency shutoff valve on its outlet.
- Nitrogen purge, then clean, the plumbing.
- It's better to start with a very clean system and keep it that way, rather than try to clean a contaminated system.
- Perform a chill-down of the plumbing and tanks.
- Never get into a Gox/Lox situation that might kill you. This is all about preparation and strict adherence to safety procedures



Part 5: High-Test Peroxide (HTP) safety (Ref.s 24 - 43)

Aspirespace has no personal experience with HTP. Users of HTP are advised to research available literature such as Ref. 24

High-test peroxide (HTP) is a high (85 to 98 percent by mass) concentration aqueous solution of hydrogen peroxide, with traces of impurities and stabilising materials such as tin salts, phosphates, nitrates, and other chemical additives. In contact with a catalyst, it decomposes into a high-temperature mixture of steam and oxygen, with no remaining liquid water.

For propulsive applications it is vital that all the peroxide decomposes into gas and no liquid remains. This puts a limit on the minimum concentration that can be used for propulsion which is 65% by mass.

Synonyms: H₂O₂, Carbamide Peroxide, Hydrogen Dioxide, Peroxide, Hydroperoxide, Urea Peroxide, Hydrogen Peroxide 100 Volumes. Physical properties

Physical properties

Hydrogen peroxide and its aqueous solutions are water-like in appearance in both the liquid and solid states. Although hydrogen peroxide is generally considered odourless, the odour of high vapour concentrations (N.B. which are harmful) has been described as sweet and comparable to the odour of weak concentrations of ozone and the halogens. Aqueous hydrogen peroxide solutions are denser, slightly more viscous, and have higher boiling and lower freezing points, than water.

Physical Parameter at standard pressure and temperature	90% Hydrogen Peroxide	Pure Water
Freezing Point, °C	-11.25	0
Boiling Point, °C	148	100
Dynamic Viscosity, centipoises	1.262	1.002
Vapour Pressure, mBar	3.45	23.4
Heat Capacity, Jkg-1K-1	2763.3	4181.8
Surface Tension, Nm-1	79.1	72.8
Density, kgm-3	1393	1000
Thermal Conductivity WK-1m-1	0.4438	0.5984

Note the high freezing temperature, this can be a problem in winter.

Note the high boiling point.

Chemically, in terms of miscibility and its action as a solvent, it behaves like water.

Since many common substances catalyse peroxide causing **exothermic** decomposition into seriously hot steam and oxygen, handling of HTP requires special care and equipment.

The use of high strength hydrogen peroxide as a rocket propellant can be traced back to Helmhuth Walter and developments in Germany during the 1930's.

Many rocket engineers tend to demonise HTP. However, German and British experience with HTP is that it is fairly benign provided sensible safety precautions are observed, and the correct clothing is used. Hydrogen peroxide is a safe and versatile chemical if handled and stored properly. With the correct materials of construction - properly prepared and conditioned - the chemical is very storable, normally decomposing at extremely low rates.



HTP can be used for regenerative cooling of nozzles where it cools as water, but only if the flow rate is high enough so that the HTP doesn't hang around long enough within the nozzle to decompose. And the HTP must not be heated above 120 degrees C, as the decomposition rate is highly dependent upon temperature.

HTP itself will not burn, but its decomposition liberates oxygen which supports combustion.

HTP is not an explosive. However, when it's mixed with organic substances at significant concentrations, hazardous impact-sensitive compounds may result. Small amounts of other materials that contain catalysts (silver, lead, copper, chromium, mercury, and iron oxide rust) can cause rapid decomposition and an explosive pressure rupture of the containing tank if it is not properly vented.

In an HTP hybrid, the HTP is passed through a 'cat pack' (a solid/metal catalyst), or is mixed with a liquid catalyst, within the combustion chamber just ahead of the fuel grain. Cleanliness is important as many contaminants - particularly phosphate - make the catalyst perform poorly ('catalyst poisoning'). The decomposed HTP contains oxygen hot enough to auto-ignite the fuel grain so a separate igniter is not required.

Decomposition

Solutions of hydrogen peroxide always exhibit a degree of instability, regardless of the concentration, continuously decomposing to form water and oxygen with the evolution of heat:

HTP (l) H_2O (l) + $\frac{1}{2}\text{O}_2$ (g) $H = 98.4$ kJ/mole

HTP (l) H_2O (g) + $\frac{1}{2}\text{O}_2$ (g) $H = 54.4$ kJ/mole

- Several factors affect the rate of decomposition, including temperature, degree of contamination, surface activity, increased alkalinity, as well as the presence of impurities and stabilisers and to a lesser extent concentration.
- The normal rate of decomposition for commercial grades of hydrogen peroxide stored in compatible equipment is extremely low, typically less than 1% loss per year.
- The ability of peroxide to coexist with a substance is called 'compatibility'.
- Peroxide is incompatible with many substances, including most of the transition metals (i.e. iron, copper, silver, cobalt, etc.) and their compounds, many organic compounds, dirt, human beings, etc.
- The use of a catalyst (such as manganese dioxide, silver, or the enzyme catalase) vastly increases the rate of decomposition of HTP.
- Smooth container surfaces are always less active than rough ones due to a lower surface area. Similarly, tanks with a lower surface area-to-volume ratio (e.g. a sphere) are less active.
- Due to slow decomposition, HTP must be stored in a vented container to prevent the build-up of pressure leading to the eventual rupture of the container.
- Any container must be made of a compatible material such as polyethylene, aluminium, or stainless steel, and be cleaned of all impurities (a process referred to as passivation: see below) prior to storing HTP in it.
- The decomposition rate of HTP depends on increasing temperature: the rate of decomposition increases approximately 2.2 times for each 10 degrees C rise in temperature in the range from 20 degrees C to 100 degrees C. So try to keep it cool!
- Commercial HTP is stabilised with various additives in order to provide a certain level of protection against the typical (low) levels of contaminants that may be experienced during transportation and storage in standard equipment.
- This stabilisation, however, is not able to protect against 'gross' contamination: don't drop something into a vat of HTP unless you like fireworks!
- Since decomposition is a significantly **exothermic** process, the temperature of the decomposing solution will continue to rise if the generated heat can't be effectively dissipated to the surroundings.



- This increase in temperature will increase the rate of decomposition resulting in a self-accelerating system or 'boil-off' which can lead to a runaway reaction.

Decomposition is usually defined in terms of AOL: the 'active oxygen loss' after some period of time from a sample of HTP at an initial concentration of peroxide (by mass). AOL can be defined as:

$$\% AOL = 100 \times \frac{(mass_i \text{ concentration}_i - mass_f \text{ concentration}_f)}{mass_i \text{ concentration}_i}$$

where subscripts *i,f* are 'initial' and 'final' respectively.

HTP stability - which is usually reported as percent stability - is the test used to determine the decomposition reaction rate of hydrogen peroxide solutions.

As applied in the materials compatibility tests, it is essentially a measure of the degree of contamination of the solution by the test material. This is determined by measuring the AOL of the HTP during a 100 degrees C exposure for 24 hours. The stability is then expressed as (100 - AOL) percent.

Corrosion

Hydrogen peroxide doesn't normally give rise to significant corrosion problems, but it can dramatically accelerate chloride ion corrosion effects on aluminium and its alloys (e.g. 6061) and stainless steel. Aluminium alloy 1060 has been found to have the lowest corrosion, and is therefore often used for pipework.

The engine plumbing shouldn't be allowed to trap HTP for any length of time after firing to prevent corrosion. Provide drain cocks at low points in the plumbing, and for valves and pumps.

Home-brew

Rocket amateurs have purchased peroxide of 70% or lower concentration (the remaining 30% is predominantly water) and increased its concentration themselves. Distillation is extremely dangerous with peroxide; peroxide vapour can ignite or detonate depending on specific combinations of temperature and pressure. In general, any boiling mass of high-concentration peroxide at ambient pressure will produce vapour-phase peroxide, which can detonate.

Passivation and cleaning

Passivation is the process of making a material 'passive' to HTP by removing the organic and catalytic elements present on the tank/plumbing wall.

All material surfaces that come in contact with HTP must be specially cleaned and treated prior to their use to minimize decomposition and material corrosion. This general process is known as 'passivation'.

In order for a tank to store HTP for extended periods it is vital that it goes through a passivation procedure.

Passivation consists of three steps:

- A chemical and physical cleaning procedure (**trike**, acetone, washing-up liquid) designed to remove oxides, scale, dirt, weld (and heat treat) slag, oil, grease, and other foreign material from the material.
- Treatment of the material with acid or alkaline solution on the surface to minimize chemical or catalytic activity between the surface and the HTP.
- The material is subjected to propellant conditioning to check the completeness of the chemical treatment and to eliminate - through further oxidation and chemical complexing - all remaining active sites.



- Normally, propellant conditioning is conducted in 35% peroxide, although many organizations prefer additional propellant conditioning of materials at the conditions (peroxide concentration and temperature) that will be experienced on firing day.

The following is a suggested passivation procedure:

	Metals		Non-Metals	
	Stainless Steel	Aluminium Alloy	Fluoropolymers	Pyrex Glass
Degreasing and cleaning	Cleaned with hot 'Decon 90' at 60°C. Scrubbed clean with nylon brush. Rinsed afterwards with distilled water to remove all traces of detergent. Handling after this stage should only occur with tongs/clean gloves.			
Descaling (Pickling)	Immersed in room temperature 2M hydrofluoric acid and 5M Nitric acid for no longer than 60 mins. After descaling rinse with distilled water at room temp.	Immersed in 0.5M hydrofluoric acid 5M Nitric acid for between 30s and 5 min at 45 °C max. After de-scaling, rinse with distilled water at room temp.	This is not applicable for polymer coatings.	This is not applicable for glass ware.
Basic passivation	Immerse in 10M Nitric acid at 20°C for a min of 30 min duration. Then rinse with distilled water and progress immediately onto the next stage.	Immersed in 10M Nitric acid at 20°C for a period of 1 hour. Then rinse with distilled water and progress immediately onto the next stage.	Immerse in 5M Nitric acid for 1 hour at room temperature. Rinse copiously with distilled water and proceed to the next stage.	Immersed in 3M Sulphuric Acid for 1 hour at room temperature. Then must be rinsed with distilled water.
Propellant conditioning	Fill with 35% Hydrogen Peroxide and leave to stand for 24 hours. Then fill with 90% HTP for 12 hours.			
System/plumbing assembly	After system integration, propellant conditioning with 35% Hydrogen Peroxide is required for 24 hours and then refilled with 90% HTP and repeated with 35% then 90% solutions.			

Note that once a material has been passivated, it won't necessarily stay passivated. Stainless steel will (preferably in a humid environment), but anodised 6061 T6 aluminium has to be stored in a nitrogen atmosphere until used with HTP.

- Items such as valves, pumps, actuators, system piping, etc. cannot be cleaned properly in the assembled state, because the solvent, cleaning solution, residual contamination etc. may be trapped in inaccessible areas. The cleaning should be done immediately before component or system assembly, unless provisions are made for packaging the passivated part (bag sealing) to protect against re-contamination until ready for assembly.
- All cleaning, passivating, and rinse solutions should be applied by immersing, spraying, wiping, circulating, or whatever so that all surfaces to be cleaned will be completely wetted and flushed with the solutions. Any section of the item to be cleaned that can trap or retain any liquid should be drained or emptied between the applications of each different solution or chemical mixture. The item should be rinsed until it is chemically neutral between each operation.
- Surfaces should not be allowed to dry off between the cleaning and the 'basic passivation' steps.
- A successful passivation is shown by no reaction of the material with the HTP (as evidenced by the lack of gas bubbles evolving from the HTP).



- If, at the end of the exposure period, the gas bubble rate is very minimal, the unexposed surfaces of the materials are cool to the touch, and the gas bubbles are not confined to a particular location, the material or part is considered acceptable. However, if rapid bubbling, clouding of solution, or a local hot spot is observed during the test, the solution should be discarded and the part re-passivated.
- Any part which shows blackening, rust streaks, or signs of excessive corrosion should be rejected.
- After the materials, parts, and/or assemblies have passed all activity checks in the selected peroxide solutions, they should be rinsed thoroughly in distilled or de-ionized water and dried with clean dry air or nitrogen.
- The water grade used - depending upon the passivation stage - should be distilled or de-ionized. Water from the South of England is seriously contaminated.
- Unless otherwise specified, all chemicals should be C.P. (chemically pure) grade or better.
- The passivation of materials for HTP service should be conducted in an area designed only for that purpose. The area must be kept clean, and free of combustible material. Use of a 'clean room' is recommended.
- Due to the chemicals being used, it is advisable to have eye-wash fountains for washing your eyes if need be.
- Passivation of components will be easier if they haven't been contaminated: when machining metals for HTP service, avoid incompatible tooling (e.g. copper, iron, carbide, non-stainless steel).
- If a part is replaced, or the plumbing modified, re-passivation is required.
- Welded assemblies should be passivated prior to final plumbing assembly.
- Once passivated, polythene-bag the components and seal the ends of pipework. Label the bags to indicate passivation. HTP plumbing is often painted green to indicate HTP service.
- Don't use Chromic acid cleaners for HTP components because chromium seriously decomposes HTP.
- After passivation, use only distilled water or clean nitrogen for hydrotesting/leak testing.

Storage

- Keep HTP away from heat, sparks, and flame.
- Store in a cool, dry, well-ventilated area away from incompatible substances.
- Store below 35°C.
- Storage of HTP should be restricted to its original shipping container or to properly designed containers made of compatible materials which have been thoroughly passivated.
- HTP that has been removed from the original shipping container should not be returned to it.
- Secondary containment should always be provided for storage vessels and the containment area must be kept clean and free from organic material.
- A properly designed, passivated container can store HTP for up to two years. Aluminium 1060 is often used for storage tanks.
- Large storage tanks should be installed within a dyke, revetment, or walls, to contain a spill (1.5 times the tank contents).
- The number of transfers of the HTP from storage tank to final use should be minimised (avoid intermediate containers) to avoid contamination.
- A spherical tank minimises tank wall surface area per internal volume to reduce decomposition. Use a small number of large tanks instead of numerous small tanks for the same reason.

Thermal runaway

Decomposition of HTP liberates heat, which eventually is released through the tank walls to atmosphere. However, the rate of heat transfer to the atmosphere increases only linearly with temperature, whereas the rate of decomposition increases exponentially with temperature.



- Consequently, for any particular storage vessel there exists a critical decomposition rate beyond which the rate of heat liberation will always exceed the rate at which heat can be transferred to the surroundings.
- Once a storage vessel passes the critical condition, a self-accelerating decomposition will set in which, unless checked, may reach a very high rate, and overpressure the tank.
- If your tank starts cooking, set up a torrent of cooling water over the tank, and then run away!
- So don't insulate HTP tanks.
- A tank temperature alarm, linked to a thermocouple inside a sealed aluminium tube or placed against the exterior tank wall below the liquid level, will warn of fire or thermal runaway.

Storage tanks should be designed with taps/drains so that HTP samples can be collected without having to insert a probe or scoop into the tank which could introduce contamination.

Don't let the HTP freeze.

Venting

Because HTP is continually decomposing - albeit normally at a very low rate - a vent pipe to atmosphere should always be provided for HTP storage and handling systems, regardless of the amount of product that is stored; never confine HTP, even in sample containers.

For normal/continuous venting, the following should be provided:

- A filtered vent for non-pressurized systems.
- A **pressure relief valve** for pressurized or vacuum systems, rated at around 45 psi gauge.

For emergency venting, a minimum of 200 cm² of venting for each tonne of 100% HTP equivalent should be provided through:

- A free lift manway (manhole) cover for tanks.
- A **burst-disc** on pressurized and vacuum vessels able to prevent the pressure rising above 10% of max allowable.

All pipelines in which hydrogen peroxide may be isolated (i.e. between valves) must be fitted with a **pressure relief valve**.

- The cavities of valves which could trap HTP when in the closed position should be vented.
- Ball valves with a drilled vent (see Lox valves) are recommended for HTP service, but globe valves are better.
- Lines and tanks should be designed to vent at the lowest practical pressure. They should not be allowed to experience over-pressurization.
- Vent outlets should be shielded to prevent dust or other contamination entry.
- It may not be practical for aerospace/space vehicle tanks to be vented, in which case the tanks must be designed to handle the eventual pressure of the liberated oxygen from decomposition.

Handling HTP

- Wash thoroughly after handling HTP.
- Remove contaminated clothing and wash before reuse.
- Use only in a well-ventilated area.
- Don't get HTP in eyes, on skin, or on clothing.
- Keep containers tightly closed.
- Avoid contact with clothing and other combustible materials.
- Don't ingest or inhale HTP.
- Discard contaminated shoes.



- Rinse empty drums and containers thoroughly with water before discarding.

For safety, at least two persons should be simultaneously involved with handling HTP so they can keep an eye on each other.

Transportation

- Use approved containers.
- Don't roll or tilt drums containing HTP or it'll spill through the vent.
- Keep the drum upright.
- Empty the drum using a pump or siphon (don't suck on the siphon!)
- Don't gas-pressurise the drum (can introduce contaminants unless great care is taken).
- Flush empty drum with clean water.
- Don't let HTP back-flow into the drum.

Light sensitivity

HTP is light sensitive. Keep it out of the light, especially direct sunlight.

Spillages

Spilling HTP on a flammable substance can cause an immediate fire.

Small spill

- Small HTP spills are easily dealt with by flooding the area with water. Not only does this cool any reacting HTP but it also dilutes it thoroughly.
- Therefore, sites that handle HTP are often equipped with emergency showers, and have hoses and people on safety duty.
- The former rocket testing establishment at Westcott had numerous outdoor baths full of warm water provided at frightening cost.
- The technical team who filled the HTP-powered SR 53 rocket fighter would routinely carry water pistols on their person.
- Dilute HTP with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container.
- Provide adequate ventilation and ample water supply for thorough flushing of accidental spillage on personnel and property.

Large Spill

- Stop the leak if you can do so without risk.
- Absorb HTP with dry earth, sand or other non-combustible material.
- Don't get water inside the fill container.
- Avoid contact with a combustible material (wood, paper, oil, clothing etc).
- Keep substance damp using water spray.
- Don't touch spilled material.
- Use water spray curtain to divert vapour drift.
- Prevent entry into sewers, basements or confined areas.
- If possible, hold the spill in a pond or diked area until the HTP decomposes.

Disposal/drainage of HTP

Normally, dilution to three percent solution (by mass) of peroxide or less should be completed before the HTP is dumped or pumped into the drainage system (which should terminate in a large body of water).

- Further dilution is required before dumping into a public water table.
- Under no circumstances should HTP be dumped into sewers or drains that lead to public water tables, unless this maximum dilution has been performed en-site.
- Internal regulations used by the Rocketdyne Division of North American Aviation, Inc. established a maximum concentration of 100 parts per million peroxide for water dumped into public water tables (note this is an old recommendation: newer legislation is probably a lot more restrictive).



- All HTP facility drainage ditches (or other spillage catch basins) should be open and lined with impervious acid-resistant concrete.
- These ditches and catch basins should be kept clean of debris and combustible material.
- The main drainage ditch should be supplied with a large water flush outlet at the highest point of HTP drainage.
- The design of the test firing area should be such that all areas are drained by gravity into the main drainage system.

Fire raising potential

Although HTP is non-flammable, it's a strong oxidiser and can readily start fires when in contact with combustible materials, particularly solid natural materials such as wood, leather, cotton, etc.

- The mechanism of fire initiation has previously been considered to be due to concentration of HTP to a critical level via evaporation of water. However, a combination of decomposition of the hydrogen peroxide and oxidation of the substrate by the HTP may be required for effective onset of a fire.
- Cleanliness of all areas where HTP is stored and handled is key to the avoidance of fires and all spills should be immediately flushed with large amounts of water.
- If fires involving HTP do occur, they should be fought with water in order to provide both cooling and dilution and because oxygen from decomposing peroxide will cause re-ignition in flame supporting environments.

Fire extinguishing media

Fires involving HTP are best controlled by using large quantities of water.

- Use water only!
- Do not use carbon dioxide.
- Do not use dry chemical.
- Make sure fire-hoses and water outlets are clearly marked.
- Hoses should be able to reach the top of the HTP fill tanks.

Vapour phase explosion

Although HTP is non-flammable, it can form explosive vapours under certain conditions (explosion occurring by decomposition of the vapour rather than combustion).

HTP vapour or mist can detonate in the presence of an ignition source. Avoid conditions that produce HTP vapour or mist, ventilate the work area, eliminate ignition sources.

This potential hazard is a function of temperature, pressure, and HTP liquid concentration. For liquid concentrations higher than 74%, explosive vapours can be generated at temperatures somewhat less than their normal boiling points.

For example, for 85% liquid HTP at atmospheric pressure, a temperature of 121 degrees C is required to generate an explosive atmosphere (compared to a boiling point of 139 degrees C).

As the pressure increases, flammable atmospheres are achieved at progressively lower concentrations of boiling liquid, until a pressure of around 4 bar is reached. At this pressure, the flammability limit of HTP vapour is 33% by weight. Above 4 bar, the flammability limit of the vapour remains constant at 33%.

Oxygen enrichment of the vapour phase

The presence of HTP enhances the normal hazards associated with flammable liquids, vapours, and gases. This is due to the propensity of HTP to generate large amounts of oxygen in combination with a significant heating effect as it decomposes.

Even if a flammable material is below its flash point and therefore normally considered to be in a safe region, the heat from decomposition of HTP could raise the material above its flash point and convert a safe system into an unsafe one.



Basically, you end up with a lot of hot Gox, creating all the hazards described in the oxygen section of this document, so read that section again.

The large volumes of oxygen generated as HTP decomposes further add to this potential problem. Oxygen enrichment greatly increases the hazards of flammable liquids, vapours and gases as follows:

- It widens the explosive limits.
- It does not affect the flash point significantly, so materials below the flash point remain safe (however see comments above). The increase is into the fuel rich area.
- It greatly reduces the energy required to produce ignition.
- It increases the rate of burning, and explosion relief becomes more difficult.
- Flame traps, flame-proofing of electrical equipment, etc. may become ineffective.

Another danger in the event of an upset is the 'mist explosion' or 'torch effect' when a mist or fine droplet spray of an organic liquid is discharged into an oxygen enriched atmosphere. Under these conditions an explosion can occur regardless of whether the organic liquid is above or below its flash point.

Oxygen enrichment can also render flammable some gases and vapours which are not flammable in air, for example ammonia and some halogenated organics.

Condensed phase explosibility without organics

(as a mono-explosive: purely on its own) (Ref. 39, Solvay Interlox, Inc.)

This refers to the ability of H_2O_2 to explode as a liquid (or solid) in the absence of organic contaminants.

- 70% HTP solutions are not explosive, even when boiling at atmospheric pressure.
- Strengths of 80% and above can be made to explode but only with difficulty.
- The current conclusion of Solvay Interlox is that 85% HTP is capable of detonation at temperatures of 45 degrees C and above, if significant initiation sources are also present.
- This energy of initiation is not judged to be realisable from normally encountered sources associated with chemical handling on a test site (e.g. water hammer, pump malfunction, impact, thermal shock...), rather an actual adjacent detonation would be needed (e.g. a mixture of 85% HTP/organic or some stronger HTP alone).
- Without an adjacent detonation as initiator, 85% HTP will not pose a detonation risk at least below 80 degrees C and perhaps not even at higher temperatures.

The question of whether an HTP vapour explosion (see above) can transmit into a liquid phase detonation has been recently considered by Solvay Interlox and it is their understanding that this event is not possible, or at worst, extremely remote.

Condensed phase explosibility with organics

HTP is a very reactive, extremely powerful oxidiser. It will oxidise virtually all organic materials, creating a serious hazard.

- Explosive mixtures can be made with organics and HTP where the strength of the HTP is less than 50%.
- Mixtures made with higher strength HTP can have an explosives power equivalent to 100% TNT, or even close to that of nitroglycerine, and approach nitroglycerine in terms of the sensitivity to shock.
- Both 70% and 85% HTP are capable of generating a detonation hazard if allowed to mix with only a relatively small amount of organic material.

HTP hazards to human health

Potential acute health effects

Contact of HTP with skin causes immediate whitening due to the production of oxygen below the skin. Extensive burns and ulcerations occur unless washed off in seconds.

- Contact with eyes can cause blindness, and so eye protection is usually used.

- Very hazardous in case of skin contact (irritant), of eye contact (irritant).
- Hazardous in case of skin contact (corrosive), of eye contact (corrosive), and of ingestion. Slightly hazardous in case of inhalation (lung sensitiser).
- Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract.
- Skin contact may produce burns.
- The seriousness of the lesions and the prognosis of intoxication depend directly on the concentration, and duration of exposure.



Respiratory system/inhalation:

- Inhalation of the spray mist may produce severe irritation of the respiratory tract, characterized by coughing, choking, or shortness of breath, **lacrimation**.
- Over-exposure by inhalation may cause respiratory irritation.
- May cause chemical burns to the respiratory tract.

Risk of chemical pneumonitis.

- May affect behaviour/central nervous system (insomnia, headache, ataxia, nervous tremors with numb extremities) and may cause ulceration of nasal tissue, and chemical pneumonia, unconsciousness, and possible death.
- At high concentrations, respiratory effects may include acute lung damage, and delayed pulmonary **oedema**. May affect blood.

Eyes:

- Inflammation of the eye is characterised by redness, watering, and itching, swelling of the eyelids. Causes severe irritation, superficial clouding, corneal **oedema**, and may cause burns. Risk of serious or permanent eye lesions, risk of blindness.

Skin:

- Inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.
- Absorption into skin may affect behaviour/central nervous system (tremor, ataxia, convulsions), respiration (dyspnea, pulmonary emboli), brain.

Ingestion:

- May be fatal if swallowed, paleness and cyanosis of the face, severe irritation, risk of burns and perforation of the gastrointestinal tract accompanied by shock.
- Excessive fluid in the mouth and nose, with risk of suffocation, risk of throat **oedema** and suffocation.
- Bloating of stomach (swells up like a balloon), belching, nausea and vomiting (bloody).



Potential chronic (long term) health effects

- HTP is toxic to lungs and mucous membranes.
- Repeated or prolonged exposure to the substance can produce target organs damage, sore throat, nose bleeds, chronic bronchitis.

First Aid Measures

Eye Contact:

- Check for and remove any contact lenses.
- In case of contact, immediately flush eyes with plenty of water for at least 15 minutes.
- Cold water may be used.
- Get medical attention immediately.

Skin Contact:

- In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes.
- Cover the irritated skin with an emollient.
- Cold water may be used.
- Wash clothing before reuse.
- Thoroughly clean shoes before reuse.
- Get medical attention immediately.

Serious Skin Contact:

- Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:

- If inhaled, remove to fresh air.
- If not breathing, give artificial respiration.
- If breathing is difficult, give oxygen. **Warning: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is HTP.**
- Seek immediate medical attention.

Serious Inhalation:

- Evacuate the victim to a safe area as soon as possible.
- Loosen tight clothing such as a collar, tie, belt or waistband.
- If breathing is difficult, administer oxygen.
- If the victim is not breathing, perform mouth-to-mouth resuscitation.
- **Warning: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is HTP.**
- If breathing has ceased apply artificial respiration using oxygen and a suitable mechanical device such as a bag and a mask.
- Seek immediate medical attention.

Ingestion:

- Rinse mouth with water.
- Dilute by giving 1 or 2 glasses of water.
- Do not induce vomiting.
- Never give anything by mouth to an unconscious person.
- See a medical doctor immediately.

Notes to Physician

- Treat symptomatically and supportively.
- Attempts at evacuating the stomach via emesis induction or gastric lavage should be avoided. In the event of severe distension of the stomach or esophagus due to gas formation, insertion of a gastric tube may be required.

- To treat corneal damage, careful ophthalmologic evaluation is recommended and the possibility of local corticosteroid therapy should be considered.

Engineering Controls

- Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapours below their respective threshold limit value.
- Ensure that eyewash stations and safety showers are proximal to the work-station location.

Protective Clothing

- Wear chemical splash-type mono-goggles and full-face shield.
- Wear rubber or vinyl coated gloves. Surgical gloves are applicable.
- Suitable protective clothing such as aprons or coveralls made of polyester acrylic fiber, polyvinyl chloride, polyethylene, terylene, dacron, dynel, vinyl, or neoprene, should be worn when handling HTP (N.B. these materials should not be treated or dyed).
- Protective 'moon suit'-style clothing that does not spontaneously absorb or combust with HTP is recommended.
- Protective clothing which lacks fire resistance, must be washed thoroughly with water should it come in contact with HTP.
- If allowed to dry in the fabric, HTP may cause a fire, particularly if the clothing is soiled.
- Wear Natural rubber boots, do not wear any form of trainer, boot, or over-boots, made of nylon or nylon blends.
- Do not wear cotton, wool or leather as the outer layer, as these materials react rapidly with HTP.
- All protective clothing should be kept free of grease and dirt, which will ignite in HTP.
- Dacron may be laundered.
- Normally, no breathing equipment is required. This is due to the very low vapour pressure of the HTP. However, if a mist of vapour is present, use a vapour respirator. Be sure to use an approved/certified respirator or equivalent.



Materials compatibility

The fire and explosion hazards inherent in HTP rocketry systems make materials selection a crucial step in designing and maintaining a safe system.

You must have an understanding of the numerous factors relating to the selection of suitable materials for HTP service: such as the material properties related to the design and operating conditions, ignition and combustion behaviour, and ease of cleaning.

Testing for compatibility (Ref. 24)

The compatibility test procedure normally consists of three basic tests:

1. An initial screening test is performed to eliminate all materials that cause gross decomposition of the HTP.



2. The compatibility of the preselected materials with HTP is then determined by measuring a rate of HTP decomposition (as percent AOL) during an immersion of the materials in HTP (of a selected composition) for a stated temperature and time interval.
3. The final evaluation in the compatibility test procedure is the determination of the stability of the HTP after contact with the material.

For details and cleaning/preparation procedures for these tests, see Ref. 24 (section 4.2.1 page 170), which is downloadable.

The following is a list of materials and their HTP compatibility. [Theoretical rating, Cole Parmer]

Material	Compatibility in 50% HTP	Compatibility in 100% HTP
stainless steel	Good	Good
ABS plastic	N/A	Excellent
Acetal (Delrin)	Severe Effect	Severe Effect
Aluminium	Excellent	Excellent
Brass	N/A	Severe Effect
Bronze	Good	Good
Buna N (Nitrile)	Severe Effect	Severe Effect
Carbon graphite	Fair	Fair
Cast iron	N/A	Good
Copper	Severe Effect	Severe Effect
CPVC	Excellent	Excellent
EPDM	Good	Severe Effect
Epoxy	N/A	Excellent
FEP fluoroplastic	Excellent	Excellent
Graphite	Severe Effect	Severe Effect
Hastelloy-C	Excellent	Excellent
Hypalon	Severe Effect	Severe Effect
Kel-F	Excellent	Good
Mylar	Excellent	Excellent
Natural rubber	Fair	Fair
Neoprene	Severe Effect	Severe Effect
Nylon	Severe Effect	Severe Effect
PFA fluoroplastic	Excellent	Excellent
PEEK	Good	Good
Plexiglass/Perspex (PMMA)	Severe Effect	Severe Effect
Polycarbonate	Excellent	Excellent
Polyester fibreglass	Poor	Poor
Polyethylene	Excellent	Excellent
Polypropylene	Good	Good
Polystyrene	Good	Good
Polyurethane	Poor	Poor
Porcelain (white)	Good	Good
PTFE (Teflon, not cloth)	Excellent	Excellent
PVC	Excellent	Excellent
PVDF (Kynar)	Excellent	Excellent
Silicone	Good	Good
Synthetic sapphire	Excellent	Excellent
Titanium	Excellent	Good
Viton®	Excellent	Excellent
Zirconium	Excellent	Excellent

Ratings - Chemical Effect:

- Excellent.
- Good - Minor Effect, slight corrosion or discoloration.



- Fair - Moderate Effect, not recommended for continuous use. Softening, loss of strength, swelling may occur.
- Severe Effect - not recommended for any use.
- N/A - Information not available.

HTP-compatible materials:

Material Type	Material Grade
Tantalum	Pure material
Polymer	Aclar
Polymer	Fluorel 2140
Polymer	Fluorel 4121
Polymer	Kel F
Polymer	Kel F 800
Polymer	Viton A
Polymer	High Density Polythene
Polymer	Vinylidon-Fluoride
Polymer	Viton B (805)
Polymer	Teflon (white)
Polymer	Teflon 100X FEP
Polymer	Teflon Tape
Polymer	Teflon Welded
Polymer	Teflon TFE
Polymer	Mylar 'A'
Polymer	Mylar 'B'
Glass	Pyrex
Ceramic	Pyroceram
Coating	Kel-F on 1060 Al

Compatibility class

Another way that materials are classified for HTP compatibility is the class system:

- Storage containers for HTP would be an example of materials considered class 1.
- Class 2 materials are satisfactory for repeated short-time contact not exceeding four hours at 72 degrees C or one week at 22 degrees C. Valves and pumps used to transfer HTP are examples of this class.
- Materials used for only short-time contact (not exceeding one minute at 72 degrees C and one hour at 22 degrees C are classified as class 3 materials.
- Class 4 materials are materials that are not recommended for use with HTP.

For example, pipework joint sealing compound and valve seals can be class 3, as their contact time with each gram of peroxide is short: not enough time for a large amount of decomposition to occur.

Aluminium

- The Aluminium alloys that have been proven compatible with HTP are all the low copper alloys (e.g. 1060 and 5652), which is in agreement with the fact that copper is a catalyst.
- The high strength structural alloys such as 2014, 2017, and 2024 are unsuitable for service with HTP because of corrosion and a high rate of decomposition of the HTP in contact with them.
- Aluminium casting alloys 43 and 356 have been employed successfully for pump and valve bodies although some corrosion generally does occur.
- Aluminium for use with HTP should be anodized if possible (anodising the inner wall of tubing is impractical), preferably with sulphuric acid, followed by a 1-hour rinse in boiling distilled water. The anodizing shouldn't contain dyes.



- Aluminium should not be polished, as polishes or contaminants could remain. Anodize instead.

HTP-compatible aluminium alloys:

Cast	40E	
Cast	43	
Cast	150	
Cast	214B	
Cast	214F	
Cast	B356	
Wrought	1060	
Wrought	1060 (99.6)	99.6% pure aluminium
Wrought	1100 (2S)	
Wrought	1160	
Wrought	1260	
Wrought	1260	Pre-treated with peroxide for 24 hours
Wrought	1360	
Wrought	3003 (3S)	
Wrought	4043 (43S)	
Wrought	5052 (52S)	
Wrought	5052 Anodized	
Wrought	5054 (54S)	
Wrought	5056 (56S)	
Wrought	5058	
Wrought	5086	
Wrought	5254 (XF54S)	
Wrought	5254	
Wrought	5254-0	
Wrought	5254-H34	
Wrought	5652-0	Requires anodising
Wrought	6061 (61S)	
Wrought	6061	
Wrought	6061 anodized	
Wrought	6063 (63S)	
Wrought	6063	
Wrought	6363	
Wrought	7072 (72S)	
Wrought	7072 (75S)	

Stainless steel

- Although there are no Class 1 stainless steels that are suitable, several Class 2 stainless steel alloys are.
- In general, the wrought or forged 300 series stainless-steel alloys with proper passivation are suitable for Class 2 service with hydrogen peroxide.
- N.B. It has been reported that some formulations of the type 303 free machining alloy are not suitable for use with HTP.
- The 300 series can be used for general plumbing and tanks, especially in place of aluminium where the presence of aluminium oxide (corrosion) can't be tolerated.
- HTP tanks have been fabricated of 347 stainless steel which contains niobium (columbium) as a welding stabilizer.
- The extra-low carbon 304 stainless steel has been shown to be an excellent HTP tank material.



- The 400 series stainless steels, whether annealed or in the heat-treated form, 40 to 58 Rc, will rust if the surface finish is greater than 10 rms (root mean square).
- Cast stainless steel is generally unsatisfactory for HTP service unless special casting techniques are followed because it can contain muck-trapping voids, and it can be subject to chromium leaching, which seriously contaminates the propellant and hastens decomposition.
- Free-machining, stainless steel alloys should not be used.

HTP-compatible stainless steel alloys:

202
301
302
304
309
310
316 (non-porous)
317
318
321
322
347
615

Other metals

In general, stainless steels suitable for HTP service - i.e. 300 series and precipitation-hardening alloys - are nonmagnetic or only weakly magnetic. Therefore, any metals that are magnetic should be suspected of incompatibility with HTP.

For example, iron, mild steel, and 400 series stainless steels are magnetic and are not suitable for HTP service. Any unknown material that is magnetic is of questionable compatibility with HTP until completely tested.

- Silicon, tantalum, tin, and zirconium are compatible with HTP. Tin has been utilised for gaskets and as solder for stainless steel.
- Most other metals are incompatible, especially silver, lead, cobalt, platinum, and iron oxide.

Surface finish

- The smoother the material finish of HTP system components, the better the compatibility.
- Finishes should not exceed 32 RMS (root mean square), and should be smoother if possible.
- This is especially important in stainless-steel storage and handling systems; a rough spot in a tank, for example, will cause HTP decomposition.

Dissimilar metals

A mixture of metals in the plumbing will cause a slightly higher decomposition rate at the interface between dissimilar metals.

Plastics

- The compatibility of plastics often is not determined by the chemical nature or composition of the polymer itself but is determined by the impurities present in it.
- Laminated plastics or compression moulded materials which contain minute pores or air pockets may be incompatible because of the accumulation of muck in these voids.
- Some authorities state that polyethylene is poor with HTP, whilst polyethylene manufacturers state that it is excellent.

- The SR53 HTP-powered rocket 'plane had polyethylene tank liners. However, polyethylene at its melting temperature (as in a fire) has ignited on contact with HTP.
- High-temperature service materials such as Kel-f and Teflon (fluoroplastics) are compatible, as is glass-filled Teflon, Mylar, and Dacron.

There are many plastic materials that break down upon extended exposure (greater than 7 days) to HTP at elevated temperatures (66 to 74 degrees C) but exhibit no effect upon 24-hour exposure.

- The majority of Viton A and Viton B compounds show this effect.
- However, most service applications are at ambient temperature conditions 10 to 50 degrees C and these same plastics and rubbers demonstrate excellent service at these temperatures: Viton A is often used for O-rings in HTP systems.

PVC plastics are permeated by HTP: they change colour. They will leach chloride ions into the HTP which will corrode aluminium. Silicone rubbers are superior, though are still permeable.

Visually inspect plastics for embedded metal particles.

Ceramics

- Glassware: very compatible - especially Pyrex (British variant) - but avoid glass with added cobalt.
- Aluminium oxide: incompatible.
- Synthetic sapphire: very compatible.

HTP is incompatible with:

Periodic Table

1a	1	2a																	3a	4a	5a	6a	7a	0
	H																		B	C	N	O	F	He
	3	4																	5	6	7	8	9	10
	Li	Be																	13	14	15	16	17	18
	11	12	3b	4b	5b	6b	7b	8				1b	2b	31	32	33	34	35	36					
	Na	Mg																						
	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36						
	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr						
	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54						
	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe						
	55	56	57	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86						
	Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn						
	87	88	89	104	105	106																		
	Fr	Ra	Ac	Rf	Ha																			

Catalyst
 Doubtful Catalyst

HTP is also incompatible with:

- Reducing materials
- ethers (dioxane, furfuran, tetrahydrofuran)
- oxidizing materials
- metals (e.g. potassium, sodium lithium, iron, copper, brass, bronze, chromium, zinc, lead, silver, nickel)
- metal oxides (e.g. cobalt oxide, iron oxide, lead oxide, lead hydroxide, manganese oxide)
- metal salts (e.g. calcium permanganate, salts of iron)
- manganese
- asbestos
- vanadium
- platinum
- tungsten
- molybdenum



- triethylamine
- palladium
- sodium pyrophosphate
- carboxylic acids
- cyclopentadiene
- formic acid
- rust
- ketones
- sodium carbonate
- alcohols
- sodium borate
- aniline
- mercurous chloride,
- nitric acid
- sodium pyrophosphate
- hexavalent chromium compounds
- tetrahydrofuran
- sodium fluoride
- organic matter
- potassium permanganate
- urea
- chlorosulfonic acid
- manganese dioxide
- hydrogen selenide
- charcoal
- coal
- sodium borate
- alkalis
- cyclopentadiene
- glycerine
- cyanides (potassium, cyanide, sodium cyanide)
- nitrogen compounds
- carborundum

HTP is caused to decompose catalytically by metals (in order of decreasing effectiveness): Osmium, Palladium, Platinum, Iridium, Gold, Silver, Manganese, Cobalt, Copper, Lead.

Explosion hazard

Many mixtures of hydrogen peroxide and organic materials may not explode upon contact. However, the resulting *combination* is detonatable either upon catching fire or by impact.

Severe explosion hazard when HTP or pure peroxide is exposed to heat, friction, mechanical impact, or caused to decompose catalytically by metals and their salts, dusts and alkalis.

HTP will react explosively with hydrocarbons (fuels).

HTP may decompose violently or explosively in contact with:

- iron
- copper
- chromium
- most other metals and their salts
- dust

Soluble fuels (acetone, ethanol, glycerol) will detonate on mixing with peroxide over 30% concentration, the violence increasing with concentration.



HTP is explosive with:

- acetic acid
- acetic anhydride
- acetone
- alcohols
- carboxylic acids
- nitrogen containing bases
- As₂S₃
- Cl₂ + KOH
- FeS
- FeSO₄ + 2 methylpyridine + H₂SO₄
- nitric acid
- potassium permanganate
- P₂O₅
- H₂Se
- Alcohols + H₂SO₄
- Alcohols + tin chloride
- Antimony trisulfide
- chlorosulfonic acid
- Aromatic hydrocarbons + trifluoroacetic acid
- Azelaic acid + sulfuric acid (above 45 C)
- Benzenesulfonic anhydride
- tert-butanol + sulfuric acid
- Hydrazine
- Sulfuric acid
- Sodium iodate
- Tetrahydrothiophene
- Thiodiglycol
- Mercurous oxide
- Mercuric oxide
- Lead dioxide
- Lead oxide
- Manganese dioxide
- Lead sulphide
- Gallium + HCl
- Ketenes + nitric acid
- Iron (II) sulfate + 2-methylpyridine + sulfuric acid
- Iron (II) sulfate + nitric acid + sodium carboxymethylcellulose (when evaporated)
- Vinyl acetate
- Trioxane
- water + oxygenated compounds (eg: acetaldehyde, acetic acid, acetone, ethanol, formaldehyde, formic acid, methanol, 2-propanol, propionaldehyde)
- organic compounds

Autoignition hazard

Most cellulose (wood, cotton) materials contain enough catalyst to cause spontaneous ignition with HTP. Also paper, oil, soiled clothing.

Welding and soldering

When welding or soldering components to come into contact with HTP bear the following in mind:

- Tin-based solder: compatible
- Lead-based solder: very incompatible



Argon arc welding: fragments of the tungsten electrode must not remain within the weld. You may need an X-ray photograph to confirm that the weld is fragment free.

Flux and carbon formed in fabrication should be cleaned from welded areas in stainless steels by a 300 series stainless steel wire brush. Any inclusions remaining should be ground out.

- For the grinding, of cast surfaces, welds, and weld spatter on surfaces that will contact HTP, a clean white aloxide (aluminum oxide) abrasive is recommended. (Weld spatter will cause decomposition.)
- The resulting smooth surface of the weld reduces decomposition.
- Note that grinding/machining/wire brushing of aluminium may cause more harm than good as it is soft.

Carborundum grinding tools must not be used.

The use of 5-percent silicon rods which are commonly used in aluminium welding are not recommended for HTP service:

- During passivation and contact with hydrogen peroxide, such welds turn black and may cause decomposition of the solution.
- If use of these rods is required, subsequent sulfuric acid anodization is necessary to stop the weld from turning black during passivation.

Inert gas welding is preferred because the welds are less porous.

- Copper electrode tips should be avoided (use stainless steel).
- Avoid 'spitting' from tungsten electrodes getting into the weld: start the arc on a piece of scrap metal and carry the weld into the workpiece.
- Similarly when stopping the arc.
- When welding stainless steel, carbide precipitation must be avoided by the correct choice of metal (stabilised or extra low carbon alloys).
- It might be best to avoid welded joints in amateur HTP rocketry plumbing unless a welding specialist is employed: rough, pitted DIY welds cause decomposition of HTP.

Filters

- Porous stainless-steel elements formed by sintering powdered metal should not be used with HTP.
- Dacron should not be used for making filters.
- When using filters for HTP, it's important to keep them clean and to examine them frequently.
- When dirt on a filter is allowed to dry, oxidation may occur which may cause increased catalytic action. Therefore, it is a good policy to flush a filter before use, and to back-wash an HTP filter immediately after use with distilled or clean water.
- Because of the massive surface area available (for promoting decomposition), the filter should be selected from Class 1 or 2 materials, and should be located where it is not constantly immersed in the liquid (such as the inlet or outlet of a transfer line).
- Also, it should be located for easy and repeated opening and cleaning.
- A 25 micron type 316 stainless steel filter can be used.

Pumps

Pumps should use compatible materials, and should incorporate a temperature alarm to warn of overheating. The pump should have a drain valve too to drain the housing after use.

Instrumentation

- Avoid contact between dissimilar metals as corrosion will occur.
- Avoid silver solder as it is incompatible with HTP (as are several other types of solder).
- Bourdon pressure gauges have internal cavities that can trap HTP and accumulate contaminants. It's best to insert an impermeable diaphragm between the HTP and the gauge.



Lap joints

Lap joints should not be used in fabrication of materials. Lap joints provide cracks, crevices, etc. (which cannot be readily cleaned), and may furnish a source of contamination; they also provide dead spaces for retention of HTP.

Lubricants

As with nitrous oxide and Lox, Use only fluorinated lubricants ('Krytox' etc).

Platings

Platings, such as tinplate or sprayed metal coatings, will blister and break off because the HTP can get in behind them.

Brazing or silver soldering

Just don't with HTP!

Hybrid design

- The hybrid HTP tanks can be pressurized with nitrogen or compressed air.
- All of the tanks and plumbing should be hydro-tested with water.

HTP in-service recommendations

- Have plenty of water on tap, such as from large elevated tanks. Conducting your firings in the middle of a lake may be going too far, but you get the point.
- Everyone to carry large water-pistols (as the SR 53 team did).
- Minimise the number of persons present in the test area whenever HTP is being loaded/is loaded into the **run-tank** or whenever HTP is flowing.
- Use a large-volume (large diameter) **burst-disc** on the **run-tank**. Mandatory for manned systems.
- Keep it very clean: even very small traces of contaminating fuel can cause major problems.
- If using HTP in a confined space, use a sensor to monitor dangerous concentrations of HTP vapour which could be inhaled.
- Properly earth all ground support equipment (fill tanks and fill lines), and keep mobile phones and laptop Wi-fi's switched off when anywhere near HTP.
- Construct tanks and lines out of conductive material. For composites, work out a way of dissipating static (aircraft composites have metal foil as their outer layer to dissipate lightning strikes. Other approach is to embed carbon nanofibres or nanotubes within the resin.)
- Prevent back-flow of igniter gas through the injector.
- Use small enough injector orifices and widen the nozzle throat to get a good pressure drop across the injector right through the burn (a drop of 20% of the combustion chamber pressure).
- Use **pressure relief valves/burst-discs** on all trapped volumes of HTP to prevent hydraulic overpressure.
- Avoid eddies or stagnation zones in the feed pipework due to sudden changes in cross-section. These can act as flame-holders which prevent any fire from being flushed safely downstream.



Part 6: additional safety issues

Engine firing safety

Firing procedures

A list of correct procedures to be carried out in a certain order should be written down in order to fire the hybrid engine (safe operating procedures). Writing a draft of these early on helps define the eventual design of the plumbing.

Test-site safety

Ensure you know where all people will be during the firing. Ensure there are clear lines of communication: you don't want anyone to not hear that a firing is just about to happen.

Designate one person as the Safety Officer, who has responsibility to ensure that the site and people are safe before, during, and just after firing.

Immediately after firing, don't all jump up and down cheering. Make sure everything is safe first, *then* jump up and down and cheer. (Or weep if it all went boom.)

Safety ethos

Safety systems including at least two barriers or safeguards should be provided under normal and emergency conditions so that at least two simultaneous undesired events must occur before any possibility arises of personnel injury, or major equipment damage.

Failure tolerance

When things go wrong - a leak appears, or the hybrid misbehaves - that's not the time to think about what to do: there usually isn't time for debate. Instead, considerable time should be spent well before firing day playing 'what if' scenarios, so that a comprehensive printed list of actions to be taken when something goes wrong are immediately to hand come the firing. These should be committed to memory.

In Aspirespace - just prior to a firing - we remind all present that if they feel something is wrong, they should shout "abort!" and the engine will be shut down immediately without hesitation or discussion. Safety is far more important than wasting propellant by shutting down perhaps in error.

Engine firing inhibits

Propulsion system engines are extremely dangerous if fired inadvertently. Several major propulsion disasters were caused by inadvertent rocket engine firings, especially on the launchpad.

As a minimum, the engine firing commands should have an electrical safe and arm inhibit on the electrical power to the engine firing drivers (igniters etc) and mechanical inhibits with safe and arm position monitoring (e.g. labelled switch positions) so that even if the monitoring signal is not functioning it is detectable. Usually, the engine firing command requires redundant signals.

There generally are two fluids inhibits on the engine, such as propellant tank isolation valves and injector manifold isolation valves in series with the engine valves. The valves - that is, the inhibits - should have indicators for valve position (an electrical signal fed back from the valve) to show closed or open for monitoring by the firing crew.

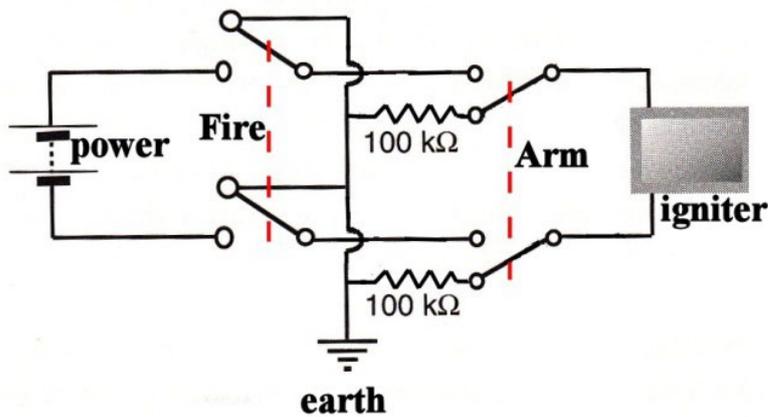
Ignition safety

Engines are generally ignited electrically; most incorporate electrical igniters.

The leads of the igniter must be shorted together right until just before launch as otherwise it can be activated by radio frequency energy (mobile 'phones, laptop Wi-fi's etc).

Take care not to create loops in the igniter leads between the short and the igniter as these can act as antennas for any rapidly changing electromagnetic fields from nearby electrical equipment, or the very rapidly changing fields from lightning storms (not necessarily a direct hit). [The more rapidly the field fluctuates, the larger the voltage induced in an inadvertent antenna.] (Ref. 1). As with solid motors, include an arming switch as well as the firing button.

The following circuit diagram illustrates a firing circuit used in industry:



Two double-pole double-throw switches; both are earthed when in the 'safe' position. Grounding the 'arm' switch grounds the igniter loop.

Because it's possible - during a lightning storm - that the rapidly changing magnetic field could create a voltage across the igniter, two very large resistors are present to reduce the current from such a voltage to a very low value.

If using a pyrotechnic igniter, make sure it's narrow enough not to block the nozzle throat if it comes loose during ignition otherwise the combustion chamber could overpressure.

Blockhouse

Rocket engines - even hybrids - can blow up the first few times until you get everything right. When they do blow up they can kill you or others who happen to be around.

Usually, the combustion chamber wall is metal; when it blows up, this turns into lots of razor-sharp red-hot fragments of metal flying through the air at several hundred kilometres an hour.

(Even if the chamber wall is made of plastic, it can still kill you if it goes bang.)

So when they're testing the hybrid, the firing team should hide behind thick brick walls (one thickness of bricks isn't enough).

They also need a specially built building with a thick roof, as large bits of blown-up hybrid might come falling out of the sky.





Any windows need glass many centimetres thick so as not to shatter. The firing team might instead use a periscope to view over the wall to protect their eyes.

A building that is tough enough to withstand a rocket explosion is termed a blockhouse.

Shown above is one of Aspirespace's former testing sites with the blockhouse in the background, the walls of which were nearly a metre thick.

The engine is fired by remote control from the blockhouse once all present are inside.

The tank pressure, chamber pressure, and the thrust are measured by electrical sensors which are connected to wires leading into a computer in the blockhouse.

Make absolutely sure nobody else can wander into the danger area while you're testing.

Engine system testing

Propulsion systems instrumentation and running/firing controls must be subjected to a rigorous verification comparable to the scrutiny of the fluid/plumbing system: there's no point having a reliable engine if its control software is ropey and unreliable.

Comprehensive system-level testing of the integrated end-to-end propulsion system, supported by qualification testing, is the preferred verification method.

It is essential that the integrated propulsion system be verified from the input stimuli (human or software commands) to the end function (engine running).

Training

The firing team must be trained to identify and respond to any hazards they might encounter in the test area. (During a firing they should of course be a long way away behind a serious amount of concrete.) This includes how to take appropriate precautions when working with hazardous materials/fluids, use of suitable protective clothing, location of any emergency equipment, and first aid techniques.

Tools

When adjusting or modifying the hybrid and its plumbing, it's important to have a written list of the correct tools to be used for each job. Tools that have become magnetised through extensive use, tools that generate high temperatures, and electrical devices, all could potentially be a hazard.

Tools used for working with an oxidiser should not come in contact with fuel or greases.

Human factors

The prevention of human error is essential, and it's important to design hardware and plan operations with this in mind: I nearly dropped a 30 kg steel combustion chamber on my foot because it had to be lifted free of its support cradle to be disassembled.

The hybrid system is to be used and serviced by humans. Don't design an engine or plumbing that is tricky or fiddly to disassemble: for example, don't force people to have to stick their hands deep into a mass of pipework or cabling where they can't see what they're doing.

This has caused accidents in the past. Keep the system user-friendly even if this costs more. Make sure there is adequate clearance around the system to allow people to work easily, and to provide ventilation.

Note: HTP decomposition depends upon surface area, so HTP plumbing should be component and surface area minimised, but not at the excessive expense of user-friendliness.

Avoid joints above head-height that could drip propellant onto people.



Keep people away from, or at least very aware of, sharp edges and protrusions, very high or very low temperature components, and high-powered electrics. Consider shields, barriers, and warning signs.

Electrical

Here are some brief electrical safety tips:

- Modern electrical safety standards are well publicised and should be used.
- Use only connectors that can't be accidentally reversed.
- Avoid earth-loops (called ground loops in America). See [http://en.wikipedia.org/wiki/Ground_loop_\(electricity\)](http://en.wikipedia.org/wiki/Ground_loop_(electricity))
- Strain-relieve all wiring: make knots or connections within the plugs connecting your wiring so that the wires can't be pulled out of their plugs if anyone trips over the wires.
- Test the whole electrical system before going to the test site.
- Don't let any of the electronics accidentally fire the engine igniter! Galvanically isolate (electrically isolate) all separate systems from the final link / safe-arm circuit and from any pyrotechnic firing circuits using opto-isolators, relays, or hall-effect current sensors as applicable.
- Use appropriate connectors and cabling. Try not to use mains connectors on low-voltage systems in case you accidentally plug them into a high voltage supply.
- Vapour-proof fixings are recommended.
- Electrical power must not be applied to the test stand/site while the system is being loaded with oxidiser.
- Earth all sections of the oxidiser plumbing. The bond to the plumbing component should have a resistance less than 10 ohms.

Data logging

Getting the signals from the pressure and thrust sensors into your computer is known as 'data logging' and is a whole book in itself on electronics.

- Try to use transducers drawing between 4mA to 20mA of current where possible.
- Try to buy sensors which are factory pre-calibrated to a known accuracy: it saves a lot of hassle.
- Mount pressure sensors so that they don't get suddenly hit with cold liquid on the sensor diaphragm (e.g. keep the pressure-tap pipe upright).
- Electrically screen the thrust-measuring load cell cables, but connect the screen at the computer end not the load cell end.
- Have one single earth lead from the test stand to the computer.
- Ensure you have lots of spare batteries and that they are all fully charged.

Software

Carefully design engine control and firing software. Identify critical commands and add safeguards such as watchdog timers, improper sequence of events detection, and requests for verification from the human operator "are you sure?"

Test site cabling

If possible, bury permanent high-voltage/mains cabling underground in rigid aluminium or steel conduits.

Test site lightning protection

All steel structures, vertical vent pipes, and large storage tanks, should have lightning protection. They should also be earthed.



Graphite nozzles

It's quite common - especially on the cooler-burning nitrous hybrids - to use graphite as an insert for the nozzle throat. Beware that graphite's quite brittle, you've got to treat it gently. If you drop it in your workshop and crack it, it'll come apart during hybrid firing.

And don't let it get damp. Graphite will happily soak up water like a sponge. When the hybrid's firing, this water flashes into high-pressure steam that expands greatly and so can split the graphite apart.

It's best to bake your graphite in the oven for an hour or two before hybrid firings to get the water out.

Fluorinated lubricants and fluoroplastics

Some fluorinated hydrocarbons will detonate on exposure to heat and aluminium. So don't use them as a lubricant for machining (e.g. on a lathe) or thread-cutting.

Some highly fluorinated lubricants are shock-sensitive in Lox, so treat gently.

Fluoroplastics and fluorinated lubricants will give off highly toxic fluorine gas when burned/vapourised so don't attempt to weld them without breathing apparatus.



Glossary:

Burst diaphragm/burst-disc: a structural fuse (a thin metal disc) that bursts at a pre-designed overpressure to unblock a pipe. These can be bought-in.

Density impulse: the product of the **Specific impulse** of the propellant (see below) times its specific gravity. Specific gravity is dimensionless, and is equal to:

$$SG = \frac{\text{propellant density}}{\text{density of water}}$$

where the density of water is pretty-much 1.0 kg/m³

A high-density impulse shows a high-performing propellant that also takes up a small tank volume, leading to a low tank mass.

Emollient: A moisturising treatment applied directly to the skin. It reduces water loss from skin by covering it with a protective film.

Exothermic: a chemical process that liberates heat.

Feed system: the pipework between the **run-tank** and the injector.

Fill-tank: the commercial container supplied with the oxidiser.

Flammability limit: Mixtures of dispersed combustible materials (such as gaseous or vaporised fuels, and some dusts) and air will burn only if the fuel concentration lies within well-defined lower and upper bounds determined experimentally, referred to as flammability limits.

Gox: gaseous oxygen.

Lacrimation: the secretion of tears; specifically: abnormal or excessive secretion of tears due to local or systemic disease or contamination.

Lox: liquid oxygen.

Oedema: also known as dropsy, is where there is an excessive build-up of fluid in the body's tissues.

Pressure relief valve: also colloquially referred to as a 'vent valve' in Lox tank applications, this is a valve that will open at a set pressure to prevent overpressure. Make sure its internal orifices are wide enough for the flow-rate required.

Run-tank: the tank inside your rocket-vehicle/test-stand that is filled from the fill tank. (In a conventional hybrid, the term 'fuel tank' is just plain wrong as the fuel is the plastic in the combustion chamber.)

Specific impulse: The number of seconds that one Newton (weight at ground level) of propellants can produce one Newton of thrust for. It's kind of like the 'miles to the gallon' fuel performance measure that applies to cars.

Stress intensity (factor): used in fracture mechanics to predict the stress state ('stress intensity') near the tip of a crack caused by a remote load or residual stresses. See http://en.wikipedia.org/wiki/Stress_intensity_factor



Trike: trichloroethylene (trichloroethene), traditional dry cleaning fluid, cleans oxygen/oxygenated systems very thoroughly. It is an anaesthetic, so wear gloves and don't inhale it. Unfortunately, trike is being phased-out because it damages the ozone layer, so alternatives are tetrachloroethylene (perchloroethylene) which is somewhat toxic (wear gloves and don't inhale it), or chloroform followed by acetone, or isopropyl alcohol. Don't let water get into the trike, as this can cause corrosion of the component being cleaned. Note that trike is flammable, as are chloroform, acetone, or isopropyl alcohol (and probably tetrachloroethylene too). Provide good ventilation when using these cleaners, or do it outdoors.

Quenching: when the heat necessary to continue the decomposition reaction is drained away into another source, stopping the reaction.



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