Field Handling of Hydrogen Peroxide

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This paper discusses handling and transfer of rocket propellant grade hydrogen peroxide. It is not intended as a step-by-step procedural document but rather a guide to safe and reasonable practices developed from comprehensive literature information, prior historical practices, and actual current field experience. Although the basic handling processes of high test peroxide is fairly scale independent, the discussions here go into the details on the differences brought about by the size of the peroxide storage container and the probable type of operation to be conducted. The intent of the paper is to introduce the new user of rocket propellant grade hydrogen peroxide to the subject of handling and transfer and thereby allow intelligent and informed decisions to be made regarding this subject matter.

Introduction

Near the end of World War II, hydrogen peroxide began to be used as a rocket propellant both in monopropellant applications and as an oxidizer in bipropellant rockets. Rather informal practices for system design and operations were used at this time yet the general success of HTP was quite good. Figure 1 shows a propellant transfer operation on the ME163 rocket plane. It should be noted that the large steam cloud probably contains HTP vapors and that the operator is not wearing reasonable PPE by modern standards.

This use was expanded through the years up to approximately 1960 to 1970 when the favor for storable propellant applications began to shift to other chemicals, such as various types of hydrazine; some as monopropellants and some as the storable fuel in a bipropellant system with nitrogen tetroxide as an oxidizer. These combinations became so popular that hydrogen peroxide basically became less attractive and used on fewer platforms. Manufacturing of rocket grade high test peroxide (HTP) diminished and it was eventually no longer used as a rocket propellant or oxidizer. In the 1990's, with the advent of "clean" non-toxic propulsion and power applications, the popularity of hydrogen peroxide began to return. As the millennium turned, a number of systems were using hydrogen peroxide, manufacturing of HTP had restarted and it seems that with the modern need for more operable and less toxic systems, HTP has come back as a viable chemical for some applications. The usage of HTP has progressively increased over the past ten years and there are now several organizations using HTP. The need to establish industry standards for the safe and effective use of this propellant are now mandatory and this paper provides information for the industry to consider and use for developing the proper methods of handling and transferring rocket propellant grade hydrogen peroxide.

Hydrogen Peroxide Transfer

The generic handling and transfer requirements for HTP are fairly scale independent, so all sizes are basically the similar at a fundamental level of safety. This initial section describes those things that are applicable for hydrogen peroxide handling and transfer at all sizes of storage and. Other sources of safety and handling information can be found in references 1, 2, and 3.

For purposes of this discussion it is going to be assumed that the storage vessels, whatever size, are vendor approved and considered safe, made from properly designed, fabricated, and passivated Class 1 materials according to approved fabrication techniques for the long term. References 4 and 5 define the criteria for material classification and also provide general criteria for the design and fabrication of Class 1 containers. Small lab-scale size containers are assumed to be roughly one gallon glass jugs furnished by a propellant vendor, passivated with a proper vented cap for long term storage of HTP. Small storage is assumed to be in standard aluminum drums which are similar to

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the historical standard US DOT specification 42D. These drums are made from Class 1 aluminum, are vented with a double head. Large scale bulk storage can be considered better quality peroxide in probably the safest vessel simply because of the large volume minimizes the ability to introduce contamination and reduces the amount of thermal loads from the environment. Bulk containment can be considered as static bulk tanks, transfer trucks or trailers as shown in figure 2 acting as temporary containment vessels, or iso-containers. Bulk handling equipment also tends to become very specific and therefore easier to control. Depending on the vessel type, the vessel might have a bottom discharge or a siphon out of the tank top.

Many of the operational sites for HTP are unfriendly to HTP. They are hot, dusty, and windy. These features aggravate the risk associated with safe operations with HTP. Despite the many negatives of these environments, HTP has been demonstrated through thousands of uses to be able to be safely and routinely handled in the worst environments one might consider. Figure 3 shows a typical hazardous test site which test 98% HTP at elevated temperatures and extremely high pressures in a basically the open environment of the Mojave Desert. The metal oxides which compose the dirt and dust of the Mojave Desert are enormously catalytic with HTP. These operations were literally conducted inside a windstorm of air born catalysts.

Personal Protective Equipment, PPE

Ordinary street clothing and some chemical protection equipment is unacceptable for use when handling rocket propellant grade hydrogen peroxide. Therefore, it is mandatory for safety reasons to wear Personal Protective Equipment, PPE when engaging in this activity. Figures 1 and 2 show two examples of PPE which may be suitable for different handling situations. Figure 4 shows the more Gore-Tex professional and costly industry standard PPE for HTP operations. Figure 5 shows a relatively low cost PVC set of PPE which is reasonable for organizations with less frequent usage of HTP.

It is recommended that Personal Protective Equipment consist of:

- Non-absorbing water resistant clothes (such as PVC rain suits or preferably specific chemical spray suits), Gore-Tex chemical spray suits suitable for use with HTP handling are available and provide comfortable protection which is extremely useful in adverse operating environments such as desert or high humidity locations. Many of the US test and operations centers can be extremely hot and or humid. This suit should be a long sleeve jacket (preferably with hood), and long pants.
- 2) Non-absorbing water repellant overshoes or boots made from PVC, nitrile, other chemically resistant materials are absolutely required. These boots should be fitted inside the pants to prevent spilled HTP from going inside the boot.
- 3) Hand should be protected with nitrile or other chemically resistant gloves. These gloves should be secured such that spilled HTP can pour inside the glove.
- 4) Hard-hat may be required based upon site requirements and should be used if a hood is not used.
- 5) Eye protection is paramount and the eyes should be at least covered by chemical spray goggles which seal onto the face and the goggle vents are covered.
- 6) Additional face protection can be provided with face shields. Some organizations consider these to be optional, but are highly recommended for additional protection and redundancy with the goggles.
- 7) While water is not part of the PPE it does provide the next greatest protection after PPE. Always have large amounts of water for near any person handling HTP for various deluging.

Protective clothes need to be non-absorbing so that the peroxide runs off rather than soaking in. If HTP soaks into absorbent materials it will begin to decompose, typically because the material is a high surface area substance and most all textile type materials have some sort of compound within them that is catalytic with HTP. These catalytic substances could be dirt, contamination, pigments, dyes, metals, etc... Once the HTP begins to react with the material it has the potential to self-distill and or auto-ignite the material. Auto-ignition of clothing with HTP will create an oxidizer supported fire which is very hot and difficult to stop. Even in the absence of a fire, the raw peroxide that does not decompose will soaks through and begins to seriously soak into the skin underneath. HTP, like water, can be absorbed into skin tissue. When HTP absorbs into skin, it reacts and decomposes creating gas bubbles. These gas bubbles stop the local blood flow in the skin and create white spots. Over a period of several minutes to hours, these white spot will go away as the bubbles of oxygen are absorbed into the body. For minor exposures, this tends to have a stinging tingling sensation and is not a serious problem. More aggressive exposures can cause blistering. Figure 6 shows a finger after exposure to HTP. Note the general whitening caused by the HTP reaction in this skin. This level of exposure is noticeably painful.

Spills of HTP onto certain materials, like leather shoes can be quite dramatic and shoes are very good candidates for catching on fire because most spills occur near hands and fall to earth, typically onto the toes of shoes. Most

shoes breathe for comfort and therefore can absorb water. Shoes are also very heavily contaminated with catalytic agents from contact with the ground. Figure 7 shows the dramatic combustion of a sneaker exposed to HTP.

The pants need to be over the top of the boots so that spilled peroxide does not go down inside the boots where it will begin oxidizing the skin of the feet or setting shoes on fire if worn under the rubber boots, an unpleasant experience. The gloves are to protect the hands and the jacket sleeves should be over the gloves to keep spilled peroxide from going down inside the gloves.

Personnel Safety

In addition to PPE, various other features and items should be used when handling HTP. Most important, copious amounts of water should be available. Water should be kept on hand and made readily available preferably in quantities of one gallon of water for every one gallon of HTP. Easy access to eyewash stations and showers should be provided. Rapid deluging of body parts, clothing such as burning shoes are best met with water in easy to access containers. Five gallon buckets of water in strategic and numerous locations can provide a fast means to mitigate many problems.

General Handling Criteria

The usage of HTP irrespective of the scale of the operation will always conform to several basic tenets. These features have been time proven and when practiced can significantly reduce risk of incidents.

HTP operations are always done in groups of two or more. HTP is never handled alone. For most operations, such as drum and bulk tank transfers, one person is physically able to perform the transfer operations. In this case, the second person ensures that the procedure are followed, monitors system parameters to make sure the transfer is safe, and provides emergency assistance in the vent of a problem. Both propel must wear PPE. This is important. If the person performing the operations is in an accident it may well be of a nature that the second person cannot respond to unless they are wearing PPE. For example if there is a large spill and the transfer person needs to get deluged, the spill may prevent the second person from approaching because it will set their shoes on fire. By the time PPE is put on, the person requiring assistance may have suffered more than necessary. PPE is not needed as much for standard operations, PPE is needed for those situations which cannot be predicted or foreseen. As such it must always be worn.

Cleanliness of operations is one of the best means to mitigate and reduce the chance of contamination. HTP systems, once clean and functional normally remain quite safe as long as proper cleanliness is maintained. Keeping systems clean and free from catalytic agents or other contaminants that can compromise the system compatibility are easy procedural steps which will make overall safety very high. Storage vessels need to be maintained clean and local transfer and handling operations areas need to be maintained clear of contaminants to reduce the risk of introducing contamination into transfer or storage equipment. Smaller storage vessels can be judiciously cleaned before transfer begins using high quality de-ionized water externally to wash off dust and other contaminants. T Note that smaller vessels may need more attention to cleanliness than the bulk storage because smaller vessels and the handling practices associated with them tend to have a greater chance of introducing contamination. Larger bulk system normally have more permanent handling equipment, such as hard lined transfer lines and dedicated transfer pumps.

In the best possible world, transfer equipment would be Class 1, however in many situations transfer equipment is Class 2. In general most transfer operations out of the primary storage container involve a degradation of the propellant which is similar top exposing the propellant to a Class 2 environment. It can be assumed that unless it is known that the transfer equipment is Class 1 that the transfer is to a Class 2 system, be it a run tank, bucket or a small beaker. This means that the peroxide will only remain in the transfer vessel for the short term, typically less than one day. A standard practice in propellant transfer with HTP is that the propellant never returns to the parent container. The reason for this is that the propellant was originally safely contained and free from contamination in the original container. Once a transfer operation is undertaken, the propellant becomes contaminated and returning the material to the parent container runs the risk of creating an unsafe storage container. In general this means that transfer from a storage tank to a system is one directional. The propellant is then either used or disposed if. This statement should put emphasis on test planning since if there is no alternative to either use or disposal, it could be financially painful to blunder the planning with a large quantity of rocket grade peroxide.

All transfer operations should have sufficient amounts of water for emergencies. This water should be enough to mitigate exposure of propellant to personnel as well as deluge spills to mitigate oxidizing hazards.

Prior to any handling operations plans should be madder in the form of written procedures for what actions will be taken for several generalized emergencies, such as exposure to personnel, spills, fires, storage vessels getting hot, tanks rising in pressure, explosions, etc...

Operations should also follow written and safety approved procedures which are designed to mitigate typical accidents such as preventing human exposure, mitigating spills, preventing propellant from becoming contaminated, etc...

The design of the transfer arrangement should ensure that there is no back flow of propellant into the storage vessel. Positive passive isolation can be used with the use of check valves, etc. All gases that are used to purge or pressurize systems should be filtered.

Filters

Filters are extremely dangerous with HTP. It is very important that when using filters with HTP the most extreme caution and attentiveness is followed as the consequences of a filter can be lethal. The function of a filter is to trap contaminants, and most contaminants are catalytic with HTP, therefore a filter will slowly turn into a catalyst bed and become very dangerous in what should be a benign part of a system, i.e. the fill system. For complex test systems, especially those using cavitating venturi, small holes, and other orifices or other contamination sensitive components, filtering the HTP may be a necessity to keep out foreign material. Many systems in the aerospace and defense communities are contamination sensitive and HTP storage vessels and transfer equipment can contain contamination, such as aluminum oxides, Teflon tape shreds, metal burrs, wire, metal oxides, etc.... If a filter is used it should be considered a high risk component not a passive part of transfer system. Additional precautions should be used to monitor the behavior of the filter such as periodic inspections, temperature measurements, flow rate or pressure drop measurements, fluid temperature changes in the filter and others. Only filter what is considered to be clean peroxide. Known-to-be-contaminated peroxide should be disposed of and never be filtered to attempt to be used since it is very possible to make a hazard which cannot be controlled.

Transfer Procedures from Storage Vessels

The generic transfer process for various storage vessels is fairly scale independent, so all sizes are similar in the fundamental considerations. This section describes detail differences brought about by scale and the equipment used at different scales.

Lab-Scale Transfer Operations from Approximately One Gallon Jugs

The typical use for this small a storage container is in supplying peroxide for sample compatibility testing and bench top tests of various sorts or very small combustion experiments, or very small systems. The receiving container is likely to be a series of small beakers or perhaps a larger transfer beaker or a laboratory instrument. Figure 8 shows a typical one gallon sample container of HTP in use in the lab-scale environment.

Because of the laboratory nature of the operations and the small amounts used, the temptation to ignore PPE is high. The fact is that PPE is probably more important in this situation than in large transfer operations because of the nature of the transfer. Obviously any spill is limited to the quantity in the jug but one gallon of HTP is a large amount if it contacts the body or is spilled into a puddle around a person. It is much easier to inadvertently get spilled on, drop and break the jug, spill when pouring out of the container, and in general the clumsiness of the operation coupled with the high likelihood that the person handling the material is not working with HTP regularly, increases the risk of an accident. Full PPE should be worn.

If the transfer method is lab bench style, then existing good laboratory practices may suffice instead of detailed transfer procedures.

There is no particular need for transfer plumbing

At a minimum, a five gallon bucket of water and at least a one gallon of de-ionized water should be immediately available for emergencies. It is also desirable to have an HTP disposal vessel available for small quantities to be put in.

Prior to transfer it is necessary to ensure that all transfer equipment (funnels, beakers, storage container) are clean by a visual inspection. This will prevent transfer of propellant into a contaminated vessel which can result in a rapid and dangerous reaction. After the visual inspection, the equipment is rinsed off with high quality de-ionized water to rinse off any surface dust or contamination or any other material which is not readily visible. Sometimes it is desirable to transfer a small amount of HTP into a system to we the surface and then dispose of this propellant to ensure that the system is completely clean. This is common practice if one were pulling a sample of HTP for chemical analysis.

Small Scale Transfer Operations with Drums

The typical use for drum storage containers is in supplying peroxide for operations with limited quantities on the order of a few gallons at a time. A test container is likely to be a small pressurized run tank or a small satellite sized vehicle. The drums are generally easily handled by two people and therefore the transfer process is quite simple.

Although the need for PPE in drum transfer is seldom questioned, it can be pointed out that, again, the danger in this kind of transfer operation is greater than in a larger bulk vessel operation because of the nature of the transfer. Drum transfer requires that the transfer equipment be installed into the drum and then removed for each operation. Transfer systems will use flexible hoses, dip tubes into open drums, pumps, and other components which increase the risk for HTP to be spilled or drained near the transfer operations. Full PPE should be worn.

In spite of the relative simplicity of the transfer, it is necessary to follow written procedures to avoid mistakes and/or omissions. Repetitive drum transfer operations can cause operators to make changes if procedures are not controlled. Drum quantity accidents are capable of spilling several gallons of propellant which will have the potential of more serious fire hazards and more dangerous personnel exposure levels.

Drums should be kept vertically upright and never laid down on their side and/or rolled around. Drum handling should be via the lifting lugs provided on the drum with a proper wheeled hoist arrangement. Small motion can be accomplished by tipping and rolling the drum on the bottom edge. Drums should be stored in a location near adequate water and with reasonable precautions against contamination. Secondary containment may be necessary and is always preferred. Figure 9 shows a set of drums in a reasonable storage environment. The drums are being stored on a concrete flat in a remote hazardous test site. In addition the drums are also being stored on top of spill trays. Protective covers on the top of the drums to reduce the amount of air born dust and dirt which can get near the drum bungs. Near the drums is a large cache of de-ionized water in the black plastic drums. Some HTP drums may be provided with secondary containment trays or over packs as shown in figure 10.

Use de-ionized water is used to clean around the cap on the drum before removing to avoid contamination entering the inside from around access opening.

These drums are made from a soft aluminum alloy and basically have no strength and therefore should never be pressurized for transfer of the contents. The best methods of transfer from a drum are siphon or vacuum transfer, pouring is not an option. The siphon method is aptly described in Ref. 1 and is an excellent method, although is probably limited to transfer into an open vessel and not a closed system. Transfer equipment is preferably Class 1 as this will introduce the least amount of contamination into the drum when performing operations. Vacuum transfer is simple and ideally suited to transfer into a closed system. It is implemented by closing off and evacuating the system and then allowing the peroxide to flow into the system via a transfer line, such as a dip tube placed into the drum with a transfer hose connecting the dip tube to the system. The mass transferred from the drum can be found from by placing the drum on a platform scale. Figure 11 shows a typical drum transfer set-up. When sufficient peroxide has been transferred, the end of the transfer line is withdrawn from the peroxide and the rest of the line is sucked dry of peroxide leaving no residual in the transfer equipment. The vacuum on the system can be pulled using gas aspirators using a clean source of gas, such as nitrogen as a working fluid. Compressed air is less desirable as this will contain entrained oil from the compressor. Oil-sealed vacuum pumps are unacceptable even with traps since any oil back streaming at all would introduce dangerous hydrocarbons into the system. Portable, flexible transfer plumbing made from corrugated Teflon tubing connected to a stiff dip tube is a simple and effective vacuum transfer system. The dip tube end of the transfer tube needs to be rinsed with de-ionized water before and after transfer and stored in a clean container. Figure 12 shows a set of drums being used with a transfer hose. Note that the dip tube on the right hand side is being stored inside a PVC protective sleeve. This sleeve protects the dip tube from contamination when not in-use to further ensure that contamination is not introduced into the drums.

It is possible that drums can have sediment in bottom sump of the drum. In general some of the drum dregs should not be used. Dip tube designs should incorporate a stand-off from the drum bottom to keep the tube from sucking residual material off the bottom of the drum. Filtering is also possible but with the constraints discussed above.

When the transfer is finished the transfer line is isolated. A good practice is not to pressurize the run tank by opening a vent valve. This will ingest the local air will entrain air bon dust and contamination into the system. Instead the system should be pressurized with a slight positive pressure with a source of clean filtered gas and then vented.

HTP drums are typically returned to the supplier emptied and rinsed with high quality de-ionized water. The specific operations to service drums prior to return are vendor specific. However, cleaned and rinsed drums are no longer hazardous as they are now very clean aluminum drums. In some cases these drums can be shipped common freight. Figure 13 shows a set of drums emptied and rinsed of HTP being prepared for shipment. Please note that these drums are on a wooden pallet which is NEVER an acceptable practice for a drum which has HTP inside.

Small leaks of HTP can contact the wood and set in on fire, heating the drums and causing further hazards. Never store drums on or near wood.

Hand pumps are less desirable as there is often too much residual left in hoses and pump arrangement that can spill around and create an undesirable and unsafe situation.

After transfer the drum should be secured and the cap be put back on.

A distinct disadvantage of transfer from drums is the relatively open hole that the transfer plumbing dip tube is inserted into. This could lead to contamination being introduced to the inside of the drum and to the peroxide from, perhaps, nothing more than airborne dust or worse. Care must be taken to minimize this. A screw-on cap over the stainless steel dip tube might be a good way to accomplish this only if the cleanliness of the cap could be maintained.

At end of an operational day, Class 2 systems should be drained of any residual peroxide which is then disposed of. After draining, vacuum transfer de-ionized water can be flushed through a system. The use of water to flush and clean HTP systems is somewhat debatable. The introduction of water into a HTP system can cause many problems and it is very important that the water used in these operations is very clean. In some case it may desirable not to flush with water to help ensure that the system is maintained clean. Both practices have been used. One of the main issues with water flushing is the risk of introducing chlorine into a system. Even very small levels of chlorine can cause major problems with Class 2 system made from stainless steel since the combination of chlorine and HTP together can attack and degrade the passivation coating, thereby rendering the system less compatible to HTP.

Bulk Tanks

The typical use for bulk storage containers is in supplying peroxide for extended testing at a more or less permanent facility with possibly large quantities of peroxide at a time or large scale systems. If a bulk system is a static tank (i.e. not a tanker trailer or an iso-container), then it will most likely be filled by a tanker truck at the point of use. Figures 14 and 15 show a tanker truck transfer operation into a bulk tank. These operations are specific to the propellant provider and are carried out by the propellant provider personnel.

Bulk tanks typically require more extensive secondary protection because of the elevated dangerous of a major spill. Therefore it is normal for a bulk container to be placed in a large secondary containment system. Figure 16 shows a secondary containment system for a large 13,000 gallon bulk tank. In a like manner to the drum storage requirements, large amounts of water are necessary to safely handle bulk HTP. Figure 17 shows the local water supply used to mitigate the hazard of the 13,000 gallon tank shown in figure 16.

The receiving container is likely to be a relatively large pressurized tank with permanent, dedicated, passivated, clean piping and valves for transfer operations. When first brought on line, this equipment needs to be statically tested with peroxide prior to use to ensure complete compatibility with the expected use. A partial fill of peroxide is put in and allowed to remain for a long enough time to ensure that the possibility of a reaction is suitably small. Unless it is known and proven that transfer equipment is Class 1 it should always be assumed that transfer equipments are at best Class 2 and treated accordingly.

Although the need for PPE is seldom questioned, the nature of a large semi-permanent facility with enclosed piping and valves staffed by day-to-day personnel causes there to be a somewhat cavalier attitude to be taken which fosters an attitude that promotes slackness more than the smaller more immediate situations. This needs to be guarded against. The need for PPE is definitely still here, generally because of larger quantities and higher transfer rates leading to the possibility of a less probable but higher consequence accident should one occur. The problem is that danger seems remote. Full PPE should be worn.

Well written and easy-to-follow procedures are absolutely necessary for this kind of operation because systems are more complex and mistakes because of repetition are more easily made. The procedures should be carefully wrung out at facility start-up to ensure that all steps are necessary and correct.

HTP is readily pumped, however pump design and materials need to be carefully scrutinized. Any pump used needs to be peroxide compatible, and one cannot generally rely completely on vendor information. Pump design and materials <u>MUST</u> be carefully examined since the material of many pump parts such as springs, packings, brazed joints, etc are not compatible with peroxide.

How ever the peroxide is transferred, some method is needed to determine the amount of peroxide transferred. A totalizing target-inline flow-meter is a good possibility. Sight tubes are also good but carefully followed procedures are necessary to ensure that the sight tube is isolated from the run tank when the tank is pressurized. Very often the run tank pressure is higher than the allowable for peroxide compatible transparent sight tubes.

Bulk operations permit the most serious spill hazards to occur with very serious threats to multiple persons. In addition, a contaminated bulk tank is a very serious situation and over pressurization of rapidly decomposing bulk storage tank is very dangerous. In some operational environments, such as the desert or tropics, temperature can

become very hot. In general a bulk tank can tolerate these conditions well as the tank will cool off at night and HTP has an enormous ability to absorb energy. A risk exists when the tank begins to deplete. The now largely empty tank can get very hot in sunlight and act as a solar collector, conducting the heat down to the puddle of HTP at the bottom of the tank. A near empty bulk tank in hot weather is potentially more dangerous than the same tank when full.

Conclusions

Know the difference between Class 1 and Class 2 and consistently follow and respect the general rules of operation and handling with HTP. Always wear and use appropriate and effective Full PPE. Maintain system and operational environments and equipment as clean as possible. Keep large amounts of water readily available. Maintain vigilance against lack of respect formed from repetitive accident free operations. No matter how much experience, the peroxide danger stays the same, be alert.

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Figure 1. ME163 Rocket Plane Propellant Transfer Operations



Figure 2. HTP Tanker Truck



Figure 3. Typical Remote Operations with HTP





Figure 5. Personnel Protection Equipment - PVC



Figure 6. Skin Bleached by HTP



Figure 7. Fire Caused by Auto-ignition of a Shoe with HTP



Figure 9. Drums of HTP with De-ionized Water Drums

Figure 8. Lab-Scale One Gallon Jug of HTP



Figure 10. HTP Drum Overpacks



Figure 11. HTP Drum with Drum Scale and Transfer Equipment



Figure 12. Aluminum Drums with Transfer Hose and Dip Tube



Figure 13. Aluminum Drums Rinsed and Ready for Return Shipment



Figure 14. HTP Tanker Truck and Bulk Storage Tank



Figure 15. HTP Tanker Truck Transfers HTP to Bulk Tank





Figure 17. Bulk Water Storage