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PACKAGE PERFORMANCE FOR LIQUID HAZARDOUS
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# PACKAGE PERFORMANCE FOR LIQUID HAZARDOUS MATERIALS IN HIGH ALTITUDE SHIPMENTS

By

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## ABSTRACT

## PACKAGE PERFORMANCE FOR LIQUID HAZARDOUS MATERIALS IN HIGH ALTITUDE SHIPMENTS

By

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Packaged products transported via the feeder aircraft network (UPS, FedEx, and USPS) are found to experience altitudes as high as 19,000 feet in non-pressurized aircraft. Packages transported on ground may experience altitudes as high as 12,000 feet when shipped over mountain passes (UPS Study, RR: D10-1013). When exposed to these conditions, products and/or packaging systems can be adversely affected by the changes in the environment.

Several types of UN certified hazardous material combination packages for liquid product were obtained from three US manufacturers in consultation with FAA. Five different test methods were developed to evaluate the various types of conditions that these packages were likely to observe in high altitude shipments. After conducting the tests, it was evident that existing test procedures used by US-DOT, UN and ICAO do not prevent leaks in air-shipped HazMat packages.

Based on the new procedures developed to test liquid hazardous packages for high-altitude shipments, it has been recommended that a simulation of high altitude shipment should include simultaneous vibration and low-pressure environment. New tests and markings on packages have been recommended for ground and air shipments of hazardous materials.

## ACKNOWLEDGEMENTS

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**Images in this dissertation are presented in color**

# 1.0 INTRODUCTION

This study investigated the effect of high altitude shipments on various forms of packaging systems, primarily used to ship liquid hazardous materials. Packaged products transported via the feeder aircraft network (UPS, FedEx, and USPS) are found to experience altitudes as high as 19,000 feet in non-pressurized aircraft. Packages transported on ground may experience altitudes as high as 12,000 feet when shipped over mountain passes (UPS Study, RR: D10-1013, 2001). When exposed to these conditions, products and/or packaging systems can be adversely affected by the changes in the environment.

In the late 1990's the Federal Aviation Administration started documenting package failures in US aircraft for hazardous materials. This data was collected based on package failure incident data that is required to be reported by shippers or carriers. The study showed a significant increase in the number of package failures of hazardous materials in commercial and cargo aircraft over a two-year period (McLaughlin, J., 2000). The results of this study are discussed in the next section.

## 1.1 Air Transport Incident Data and Analysis of Packaging Failures

Incident data from the US Department Of Transportation's Hazardous Materials Information System was analyzed for 1998 and 1999. The analysis on packaging failures focused on declared dangerous goods shipments. A review of the 1998 and 1999 packaging failure data showed that a failure of inner containers in combination packaging contributed to 1299 spills or leaks. Of the incidents involving glass containers, 50% of the incidents were attributable to dropping of the packaging and failure of the inner receptacle. The information for incidents reported in air transportation were the only ones considered in this analysis. The purpose of the analysis was to try and identify any trends that could be used to enhance transport safety by reducing the number of incidents in air transportation. This was attributed to the crash of the ValuJet Flight 592 in Florida in May 1996 that was found to be a result of improper packaging and placement of unmarked oxygen canisters. The various issues involved in this incident are discussed in Section 1.2.

The incidents reported for 1999 were reviewed for package failures and the data compared to the 1998 data for resulting trends. The packaging failures were reviewed from three different perspectives to determine if any particular causal factor was skewing the results. The following issues were considered:

- Overall packaging failures
- Failures by commodity
- Failures by carrier

The packaging failures for 1998 were categorized into eight different causal factors. Two additional causal factors for 1999 were added. These were the "apparently dropped" category, which was previously counted as dropped, and the "inner container broken" category, which is a more specific definition than "unknown". Neither addition of the more specific categories in 1999 analysis significantly affected the comparisons of the two years. The packaging failures in

1999, were considered to have been caused by the following ten different causal factors:

- Seal/Closure
- Unknown
- Inner Container Broken
- Punctured
- Forklift
- Seam
- Other Freight
- Chime
- Dropped
- Apparently Dropped – this includes packages, which were found to be leaking

Table 1 shows a summary of all packaging failure incidents that were observed in 1998 and 1999. Table 2 shows the difference of failures in combination packaging, that met the existing DOT HazMat specifications and were identified and those that were unmarked. Almost 50% of the specification combination packaging experienced failures. Table 3 describes the four most frequent package failure types. The Plastic/4G showed the highest numbers of package incident failures. This category also showed the largest increase in package failures between the two years (72%).

**Table 1: Packaging Failure Incidents Observed in 1998 and 1999**

| Packaging Type | 1998 | 1999 | Change |
|---|---|---|---|
| Combination Packaging | 597 | 692 | +95 |
| Single Packaging | 231 | 243 | +12 |
| Unknown | 45 | 28 | -17 |
| **Total** | **873** | **963** | **+90** |

**Table 2: Combination Packaging**

| Packaging Type | 1998 | 1999 | Change |
|---|---|---|---|
| Specification Combination Packaging | 298 | 392 | +94 |
| Combination Packaging (Unknown Specification) | 299 | 300 | +1 |
| **Total** | **597** | **692** | **+95** |

**Table 3: Specification Combination Packaging: Four Most Frequent Failures**

| Package Type | 1998 | 1999 | Change |
|---|---|---|---|
| **Plastic/4G** | 100 | 172 | +72 |
| **Metal/4G** | 71 | 67 | -4 |
| **Glass/4G** | 46 | 64 | +18 |
| **Unknown** | 19 | 30 | +11 |

The study further analyzed the causal factors attributed to each type of package failure. These are shown in Table 4. The results showed that the Seal/Closure factor was the most frequent cause of failure in all package types.

**Table 4: Factors Contributing to Specification Combination Packaging Failures**

| Packaging Type | Seal Closure | Unk | Inner Broken | Punc -tured | Fork Lift | Seam | Other Freight | Chime | Drop | Possible Drop | Total |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Plastic/4G | 65%<br>111 | 3%<br>5 | 1%<br>2 | 2%<br>4 | 3%<br>6 | 0 | 5%<br>8 | 0 | 9%<br>16 | 12%<br>20 | 172 |
| Metal/4G | 66%<br>44 | 4%<br>3 | 0 | 4%<br>3 | 6%<br>4 | 4%<br>3 | 0 | 0 | 7%<br>5 | 7%<br>5 | 67 |
| Glass/4G | 23%<br>15 | 6%<br>4 | 17%<br>11 | 0 | 5%<br>3 | 0 | 1%<br>1 | 0 | 22%<br>14 | 25%<br>16 | 64 |
| Unk/4G | 27%<br>8 | 33%<br>10 | 0 | 0 | 13%<br>4 | 0 | 7%<br>2 | 0 | 13%<br>4 | 7%<br>2 | 30 |

Table 5 shows the four most common failures in the packages that did not meet the existing DOT HazMat specifications.

**Table 5: Unknown Specification Combination Packaging: Four Most Frequent Failures**

| Package Type | 1998 | 1999 | Change |
|---|---|---|---|
| Plastic/Fib | 98 | 85 | -13 |
| Metal/Fib | 66 | 53 | -13 |
| Glass/Fib | 73 | 50 | -23 |
| Unk/Fib | 23 | 38 | +15 |

Table 6 describes the data showing the various causal factors attributed to the unknown specification packaging failures. Again, the Seal/Closure factor was the highest contributing cause of package failures. The highest failures were found in Metal and Plastic primary containers that were packaged in fiberboard corrugated shippers as part of the "Combination Package".

**Table 6: Factors Contributing to Unknown Specification Combination Packaging Failures**

| Pkg Type | Seal Closure | Unk | Inner Brk | Punc -tured | Fork Lift | Seam | Other Freight | Chime | Drop | Possible Drop | Total |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Plastic /Fib | 62% 53 | 18% 15 | 2% 2 | 0 | 2% 2 | 5% 4 | 3% 3 | 0 | 4% 3 | 4% 3 | 85 |
| Metal /Fib | 70% 37 | 11% 6 | 0 | 0 | 2% 1 | 6% 3 | 0 | 0 | 9% 5 | 2% 1 | 53 |
| Glass /Fib | 26% 13 | 20% 10 | 26% 13 | 2% 1 | 2% 1 | 0 | 0 | 0 | 16% 8 | 8% 4 | 50 |
| Unk /Fib | 58% 22 | 37% 14 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 5% 2 | 38 |

There was an overall increase in the number of combination packaging failures. Plastic inner containers failed more frequently than any other inner packaging for both 1998 and 1999. Plastic inner packaging failed in approximately

27% of the 1999 incidents reviewed, and 23% in 1998. Closure failures were attributed to over 50% of all the package failures (spills or leaks). When glass inner containers were used, the inner packaging broke in over 50% of the instances, as a result of the container being dropped.

## 1.2 ValuJet DC-9, Flight 592

On May 11, 1996, at 14:13 eastern daylight time, a Douglas DC-9-32 crashed into the Everglades about 10 minutes after takeoff from Miami International Airport, Miami, Florida. As a result, the pilots, flight attendants, and all 105 passengers were killed. Visual meteorological conditions existed in the Miami area at the time of the takeoff. Flight 592, operating under the provisions of 14 CFR Part 121, was on an instrument flight rules flight plan destined for the Hartsfield International Airport, Atlanta, Georgia.

The National Transportation Safety Board (NTSB) determined that the probable cause of the accident, which resulted from a fire in the airplane's class D cargo compartment, was initiated by the actuation of one or more oxygen generators that were being improperly carried as cargo (http://www.ntsb.gov/publictn/1997/AAR9706.htm). The following were the summarized causes of the accident by the NTSB:

- The failure of SabreTech to properly prepare, package, and identify unexpended chemical oxygen generators before presenting them to ValuJet for carriage

- The failure of ValuJet to properly oversee its contract maintenance program to ensure compliance with maintenance, maintenance training, and hazardous materials requirements and practices; and
- The failure of the FAA to require smoke detection and fire suppression systems in class D cargo compartments

Investigators believed that no action was taken on a previous incident that occurred in an American Trans Air DC-10 plane in Chicago in 1986 that was carrying oxygen generators improperly placed in a forward cargo compartment. The plane was still on the ground when a fire started, and the airplane was destroyed. Fortunately no one was killed during this earlier incident.

Since the ValuJet incident, the Federal Aviation Administration is spending more time and money ensuring that those responsible for shipping of dangerous goods on aircraft follow the rules. This is a tough challenge since it involves participation and responsibility between shippers, freight forwarders, and air carriers and has many commercial airlines wondering if they should be in the dangerous goods business at all.

Both the regulations and enforcement are tightening. The training costs are escalating. The insurance costs are astronomical. The potential fines are staggering. The repercussions can be devastating, even deadly. The FAA now has five times the HAZMAT staff it did before ValuJet 592, and has collected more than $14 million in fines between 1997 and 2000. Although the FAA's crackdown is aimed mostly at shippers and commercial carriers, it's important to realize that the same HazMat rules apply to each and every individual passenger who is flying with accompanying luggage. Since September 11, 2002 the level of threat on commercial airlines has increased. There is an increased level of inspection in flights for unmarked/unidentified packages. Figure 1 shows the various stowage locations on an aircraft that are used to place packages, that may contain hazardous materials (www.ngwrc.org/Dulink/DU_Counterweights.html).

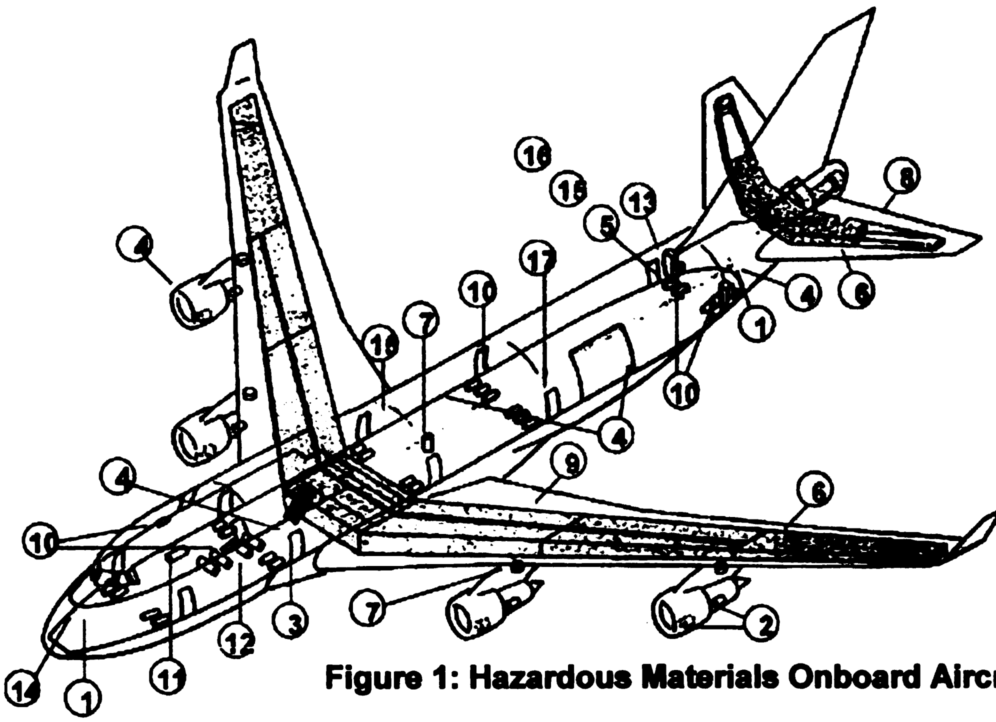


**Figure 1: Hazardous Materials Onboard Aircraft**

| | | | |
|---|---|---|---|
| 1 | **Batteries, Aircraft** (qty 2) | 10 | **Oxygen Bottles, Portable, Gaseous** |
| 2 | **Engine Oil** (waste only) | 11 | **Oxygen Bottles, Crew System, Gaseous** |
| 3 | **Escape Slides/Life Rafts** (all entry doors/rafts optional) | 12 | **Oxygen Bottles, Passenger System, Gaseous** (standard) |
| 4 | **Fire Bottles** (APU, engines, lower cargo compartment, and lavatory waste containers) | 13 | **Oxygen Generators** (optional: each PSU, standard: each attendant station and lavatory) |
| 5 | **Fire Extinguishers** (attendant stations, closets, galleys, etc.) | 14 | **Rain Repellant** |
| 6 | **Fuel** | 15 | **Refrigerant** (located in each galley) |
| 7 | **Hydraulic Fluid, reservoirs** (waste only) | 16 | **Smoke Hoods** |
| 8 | **Uranium** (depleted, counter-balance weights) | 17 | **Tritium Signs (aisles, emergency door exits)** |
| 9 | **Ordnance Devices** (off-wing escape) | | |

A feeder aircraft provides a service by connecting at least two ports in order for the freight (generally containers) to be consolidated or redistributed to or from a hub in one of the ports. The distances covered by a feeder aircraft are usually about 500 miles. A feeder aircraft fleet usually consists of propeller aircraft that do not have pressurized cargo holds. This means that the air pressure inside a feeder aircraft is the same as whatever altitude they fly at. This has a direct impact on the distribution environment for any packages the feeder aircraft may carry in their cargo holds. Figures 2 and 3 are examples of the feeder aircraft used by FedEx.

**Figure 2: Caravan Super Cargomaster Aircraft**

Federal Express depends on a fleet of more than 300 Caravan Super Cargomasters to feed overnight packages to and from outlying communities.

In cargo configuration, the Caravan offers 452 cubic feet of cargo space, including the standard cargo pod. That makes it the largest cargo-dedicated single-engine turboprop built today (http://caravan.cessna.com/)

**Figure 3: F27-500 Aircraft**

Cargo and commercial aircraft have a pressurized cargo hold, usually maintaining pressures of 8,000 feet. Figures 4 and 5 are examples of cargo aircraft used by Federal Express.


**Figure 4: McDonnell Douglas DC-10-10CF**

**Figure 5: Boeing MD-10-10F**

The existing HazMat package shipments are enforced by the Department of Transportation under the CFR 49. These specifications describe the various tests that need to be conducted on packages used to ship dangerous goods. In the existing test methods there is no differentiation between ground and air shipments. In a preliminary study Singh and Burgess (2001) found that certain pre-approved HazMat packages when exposed to simultaneous low pressure and vibration resulted in leaks. Based on this study the Consortium for Distribution Packaging at MSU initiated a study to investigate the cause of failures in current approved DOT packages and to develop new test methods to qualify all HazMat packages for air shipments. The funding for this research was provided by the Federal Aviation Authority, the Department of Transportation, and Federal Express.

At the initiation of this study, it was known that the existing DOT specifications for liquid HazMat packages did not **specifically** require the **vacuum** test. The existing pressure differential allowed users to choose between a vacuum, internal pressure test or hydrostatic test. The theoretical model developed in Chapter 3 showed a reduction in seal force based on the combined vibration and vacuum environment. The hypothesis was based on conducting tests using the combined vibration and vacuum method used in the models, and show the presence of leaks based on a reduction in seal force.

The objectives of this study were:

- Investigate current approved (DOT and UN) HazMat combination packages for liquids when subjected to simultaneous vibration and pressure levels found in commercial and cargo air shipments.
- To develop and validate a lab test method to simulate high altitude air/truck shipments of Hazardous Materials.
- To evaluate marking requirements for air-shipment of Hazardous Materials.
- To investigate effect of seal integrity and removal torque on current approved (DOT and UN) HazMat packages.
- Provide recommendations to FAA and DOT for safe shipment of hazardous materials.

# 2.0 LITERATURE REVIEW

A hazardous material is defined as "a substance or material including a hazardous waste, a hazardous substance, a material transported at an elevated temperature (hot), a marine pollutant, or any material meeting the definition for the hazard classes or divisions found in part 173 of 49 CFR, which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated". ( § 171.8, 49CFR).

A simplified definition of a "hazardous material" is any material which is known to create a danger to any person's health, life, or property through contact, exposure, inhalation, fire, explosion, or which could cause environmental pollution. These materials include fuels such as gasoline, propane, and fuel oil, household cleansers, pesticides, herbicides, drugs, medicines, paints, inks, fertilizers, as well as explosives, industrial chemicals and radioactive materials. Under certain conditions, and in large quantities, even alcoholic beverages and some foodstuffs may pose hazards in transportation and are therefore regulated.

## 2.1 Classification of Hazardous Materials

The Hazardous Materials Table (49 CFR Subpart B, §172.101) designates the materials listed therein as hazardous materials for the purpose of transportation of those materials. For each listed material, this table identifies the hazard class or specifies that the material be forbidden in transportation, and gives the proper shipping name, or directs the user to the preferred proper shipping name. In addition, this table specifies or references requirements pertaining to labeling, packaging, quantity limits aboard aircraft and stowage of hazardous materials aboard vessels. The ten columns provide information on:

- Mode of transportation restrictions/conditions
- Proper shipping name
- Hazard class
- UN or NA identification number
- Packing group
- Required labels
- Special provisions
- Packaging requirements
- Air transportation
- Vessel transportation

Appendix A shows the description, proper shipping names and markings for hazardous materials.

## 2.2 Package Design Qualification Testing

Currently, design qualification testing is performed to determine the capabilities of a packaging (CFR 49, Subpart M, §178.600). The following are the required tests for POP (Performance Oriented Packaging) for shipment of hazardous materials:

### 2.2.1 Conditioning

Packages need to be conditioned under appropriate test requirements. The various conditioning environments recommended include:

- Standard conditioning: 23 ± 2°C (73 ± 4°F)
- High Temperature Conditioning: 40 ± 2°C (104 ± 4°F)
- Low Temperature Conditioning: -20 ± 2°C (-4 ± 4°F)

### 2.2.2 Drop Test

The packages are tested to prevent Hazardous Materials from leaking or escaping if the package is dropped during conditions of transport. Packages as prepared for transportation are dropped from the appropriate height onto a rigid, horizontal and flat surface. The number and type of drops depend on the packaging being tested. The drop height will depend on the Packing Group and Specific Gravity of the material for which the packaging may be used.

### 2.2.3 Stack Test

This test ensures the ability of the packaging to remain intact and hold its contents under normal stacking conditions during transport. Test samples are subjected to a force applied to the surface of the sample equivalent to the total weight of identical packages that may be stacked on it during transport. The minimum stack height is no less than 3 meters (10 ft.) for all packages.

### 2.2.4 Hydrostatic Pressure Test

This test ensures that the packaging will not leak under pressure. Packaging systems to be tested are filled with water or other suitable liquids so as to eliminate all air pockets. The appropriate amount of pressure is applied internally through a fitting that has been installed on the packaging for this purpose. The pressure must be maintained for 5 minutes for metal and composite glass, porcelain, or stoneware, and for 30 minutes for plastic and composite packagings made of plastic material.

### 2.2.5 Leak Resistance Test

This test ensures that the package will not leak or permit liquids to escape. The receptacle is subjected to a low positive internal air pressure and, by immersion or other means, the presence of any leaks is detected.

### 2.2.6 Vibration Test

In addition to the above tests, non-bulk packagings must be capable of withstanding the vibration test specified under §178.601. The packaging is placed on a vibrating platform and restrained from horizontal movement, but free to bounce, rotate, and move vertically. The test is performed for one hour and at a frequency that causes the package to be raised from the platform in such a manner that a piece of material such as steel strapping or paperboard can be passed between the bottom of the package and the platform. After the test, the package is checked for leaks.

### 2.2.7 Cooperage Test

The cooperage test shall be performed on bung type wooden barrels prior to initial use and, where specified, at periodic intervals. The hoops of the barrel above the bilge are removed and the tendency of the staves to straighten is measured.

### 2.2.8 Periodic Retesting

Periodic retesting must be done at intervals of sufficient frequency to ensure that the packaging produced by the manufacturer is capable of passing the design qualification tests. For single or composite packagings, the periodic retest is to be done no less than once each 12 months. For combination packagings the retesting must be done no less than once each 24 months. The requirements of the periodic retest are the drop, leak-proofness, hydrostatic pressure, and stacking tests.

## 2.3 Air Eligibility Package Marking

The inability of air transport operators to discern between packaging qualified for air transport and a lack of shipper awareness of additional air packaging requirements is resulting in unauthorized or improper packaging being offered and accepted for air transport. The ICAO (International Civil Aviation Organization) Technical Instructions (ICAO-TI) includes general requirements applicable to combination packaging that are not required by other modal regulations (e.g. the requirements in 4;1.1.4, 4;1.1.6 and 4;1.1.10). Packagings prepared for road, rail or sea transport are not required to be capable of withstanding without leakage a vacuum test, be packaged with absorbent material, and subjected to a positive means of retaining friction-type closures and more stringent inner packaging quantity limitations. These additional air transport requirements may be overlooked given that current UN packaging certification markings for combination packaging do not provide an indication of whether additional air transport requirements have been met.

Investigation has shown that packagings that are not in compliance with air transport requirements frequently enter the air transport environment and that some shippers are either unaware of the additional air transport requirements or are ignoring them. Incident data show that leaking inner receptacles of combination packaging represent a significant percentage of the packaging failures in air transport (http://www.iata.org/events/dg/_files/McLaughlin.pdf). Some air mode packaging failures result from packagings that are not air eligible, but appear so because of a lack of distinguishing marks. Without an effective means of identifying packagings that meet the air transport requirements, air operators and inspectors can not easily determine if a packaging is qualified and safe for air transport.

While there is no regulatory requirement that a marking be applied to indicate that a packaging is eligible for air transport, several packaging vendors and military shippers are voluntarily marking packagings that meet the additional air requirements through the use of an icon/symbol (i.e. an airplane silhouette inside a circle) or a statement such as "Air Eligible". Use of a marked symbol or statement would be beneficial for indicating that a package is qualified for transport by air and could heighten shipper awareness and responsibility for meeting air transport requirements.

The Dangerous Goods Panel (DGP) is proposing to adopt a new paragraph 4;1.1.20 as follows:

"Combination packaging must be marked to indicate that the packaging meets the applicable requirements of this part, particularly those applicable only to air transport (e.g. the relevant packing instruction requirements, pressure differential test, requirement to provide absorbent material and closure requirements). The marking must be durable, legible, of such a size relative to the packaging as to be readily visible and placed adjacent to the markings prescribed in 6;2.1.1. The marking must include the words "Air Eligible" and/or the symbol:

## 2.4 Package Shipping Orientation

The existing DOT specification for shipping and handling HazMat containers requires that the packages be placed with the closure facing up at all times. While this practice can be easily followed for ground shipments, it is difficult to control in air transport. Air shipments are generally cubed out and therefore packages are placed in the orientation most likely to provide high cube efficiency. In various studies (Singh, et-al, 1996; Newsham, et-al, 1999) there is a clear indication that single parcels get exposed to impacts and vibration in all orientations during parcel handling, sorting, and transportation.

This study evaluated the performance of HazMat packages in the sideway and top-down orientation.

# 3.0 MATHEMATICAL MODELS

The existing test procedures used by DOT for testing and approving combination packages for HazMat shipments are sequentially performed. In addition to testing for the physical forces, a pressure differential test is required. The pressure differential test requirement can be met by either the hydrostatic pressure test or a vacuum test and this can be a problem. This section shows the mathematical models of packages and closures subjected to various conditions of high altitude shipments.

## 3.1 Analysis of closures at high altitude:

**3.1.1 Situation A**: Forces on cap and liner during application torque at ground level are shown in Figure 6.

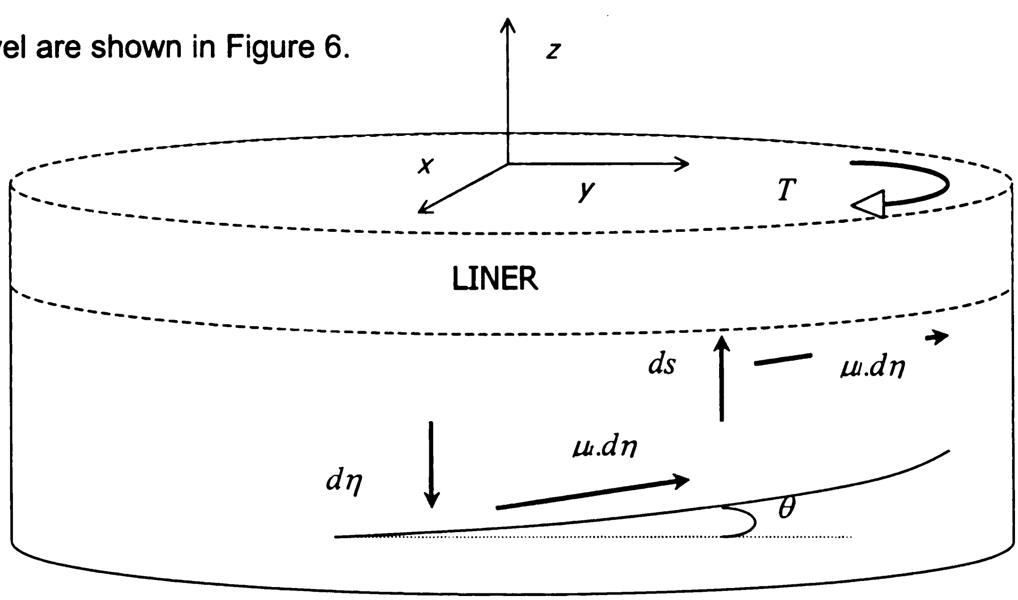



**Figure 6: Free Body Diagram of Closure at Ground Level**

Where,

$T$ = application torque (in-lb)

$d\eta, ds$ = continuous distribution of infinitesimal contact forces on thread and liner respectively (assumed uniform)

$\mu l$ = coefficient of friction between the liner and rim of the bottle

$\mu t$ = coefficient of friction between the bottle and cap threads

Since all the $d\eta$'s and $ds$'s are assumed to be the same, the following equilibrium equations are automatically satisfied by symmetry:

$$\sum F_x = 0 \quad (1)$$

$$\sum F_y = 0 \quad (2)$$

$$\sum M_x = 0 \quad (3)$$

$$\sum M_y = 0 \quad (4)$$

Let $S$ be the total force exerted by the rim of the bottle up on the liner. Let $N$ be the total force exerted by the threads of the bottle down on the threads of the cap. Then, since the thread pitch angle $\theta$ is small, we get:

$$S = \sum ds \quad (5)$$

$$N = \sum d\eta \quad (6)$$

Balancing the vertical forces and torques around the bottle axis gives

$$\sum F_z = \sum_{\infty} ds - \sum_{\infty} d\eta = 0 \quad (7)$$

$$\sum M_z = T - \sum(\mu t.d\eta.R) - \sum(\mu l.ds.R) = 0 \quad (8)$$

where $R$ = cap radius. Equation (7) and Equation (8) give

$$S = N \quad (9)$$

$$T = \mu t.N.R + \mu l.S.R \quad (10)$$

Substituting $N = S$ and $D = 2R$ in Equation (10), we get

$$S = \frac{T}{\bar{\mu}.D} \quad (11)$$

where, $S$ = seal force (lb)

$D$ = cap diameter (in)

$T$ = application torque (in-lb)

$\bar{\mu} = \frac{(\mu_t + \mu_l)}{2}$ = average coefficient of friction

As an example, consider a wide mouth bottle with a diameter $D = 3$ inches. The application torque can be anything, but is usually determined by the following industry rule ("Closure Guide", Closure Manufacturers' Association): the application torque should be about half the diameter of the cap in millimeters. Since $D = 3$ inches $= 76.2$ mm, $T$ should be about $76.2/2 \approx 38$ in-lb. The average coefficient of friction will be taken as $\bar{\mu} = 0.2$ ("Model for Predicting Application Torque and Removal Torque of a Continuous Thread Closure", Supachai Pisuchpen, 2000). Then, from Equation (11),

$$S = \frac{38}{0.2x3} = 63 \text{ lbs} \quad (12)$$

The seal force is important because it directly relates to the ability of the liner to contain the contents of the bottle. It is literally the force holding the liner down against the rim of the bottle. The larger the seal force, the greater the seal. This is not the way the industry typically looks at seal integrity, however. It is normally quantified by the removal torque.

In the simplified force diagram in the figure in Situation A, the torque and friction forces will be reversed during removal. The equilibrium equations would then give the same relationship as in Equation (11) between removal torque and seal force. The analysis therefore predicts that the removal torque is the same as the application torque. In reality, it is usually less. The reason is that the pitch angle, $\theta$ (assumed zero in the analysis) does affect the force balances somewhat. Including it in the analysis does predict that the removal torque is somewhat less than the application torque ("Model for Predicting Application Torque and Removal Torque of a Continuous Thread Closure", Supachai Pisuchpen, 2000). Regardless of the details, there is a direct relationship between removal torque and seal force, so there is no reason to question the industry practice of using removal torque to evaluate seal force.

From Equation (11) it can be seen that increasing the cap diameter decreases the seal force because less friction and therefore less seal force is required to balance the application torque using a larger moment arm. Increasing the coefficient of friction decreases the seal force because more of the application torque is spent in overcoming friction. Increasing the torque increases the seal force. But if the application torque is chosen according to industry practice (half the cap diameter), then

$$T = 12.7 * D \tag{13}$$

$T$ = application torque (inch-lbs)

$D$ = cap diameter (inches)

Substituting this in Equation (11) gives,

$$S = \frac{12.7}{\overline{\mu}} \tag{14}$$

$S$ = seal force (lbs)

$\overline{\mu}$ = average coefficient of friction

Equation (14) says that following the industry rule leads to a seal force which is *independent* of the cap diameter. From the point of view of engineering stress analysis (S.P. Timoshenko, *History of Strength of Materials*, McGraw Hill, New York, 1953), this practice would not be advisable because the *stress* on the liner would diminish as the cap diameter increases,

$$\text{stress} = \frac{S}{\pi . D . t} = \frac{12.7}{\pi . \overline{\mu} D . t}$$

$\overline{\mu}$ = average coefficient of friction

$D$ = cap diameter (inches)

$t$ = thickness of rim of bottle (inches)

stress = compression stress on liner (psi)

A smaller stress produces a smaller strain and so the industry rule would lead to larger liners being compressed less. The potential for leaks, therefore, increase as the cap size gets larger.

### 3.1.2 Situation B: At altitude (pressure differential effects)

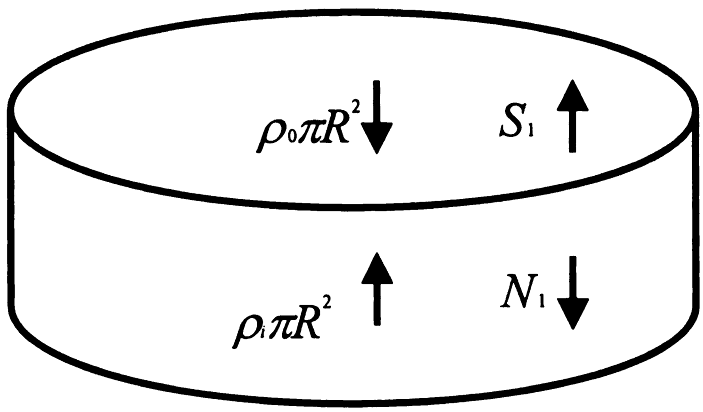



**Figure 7: Free Body Diagram of Closure with Pressure Differential**

The free-body diagram in Figure 7 shows the forces on the cap and liner after the application torque has been removed and when the sealed container is subjected to an air pressure differential.

There are friction forces acting on the cap that are not shown in Figure 3. The friction forces on the liner and friction forces on the threads are equal in magnitude but act in opposite direction, so they balance each other and therefore do not enter any force or torque balance equations. The outside air pressure, $\rho_o$, due to transporting the container by plane decreases with increasing altitude and the inside air pressure, $\rho_i$, remains constant at whatever the pressure was for the elevation the container was sealed at. The only equilibrium equation that changes is:

$$\sum F_z = \rho_i.\pi.R^2 + S_1 - \rho_o.\pi.R^2 - N_1 = 0 \quad (15)$$

which gives, $N_1 - S_1 = \pi.R^2(\rho i - \rho o)$ (16)

The thread and seal forces are therefore no longer the same. Since the outside pressure is less than the inside pressure, the thread force is greater than the seal force.

Assume the cap and bottle to be rigid (i.e. when the pressure differential is applied, there is no deformation of the cap or bottle). In this case, since the cap and bottle do not deform, and there is no pressure change inside the bottle, there is no change in the spacing between the top of the cap and the rim of the bottle, and hence no change in the compression of the liner. This means that there is no immediate change in the seal force, although the seal force will decrease over time because the liner material relaxes. Therefore,

$S_1 = S =$ same as when application torque was removed and

$$N_1 = S + \pi.R^2(\rho i - \rho o) \quad (17)$$

Hence, an increase in altitude will cause an increase in the thread contact force of magnitude $\pi.R^2(\rho i - \rho o)$, but no change in the seal force. These conditions persist for as long as the pressure differential does.

To get an estimate of how large this increase is, the following general rule, based on published pressure versus altitude charts, will be used: for every 1000 feet of altitude, the pressure drops 0.5 psi. This is considered to be the normal lapse rate in aviation. Then,

$$\rho_i - \rho_o = 0.5A \qquad (18)$$

where $A$ is the altitude above the location at which the bottle was capped, in thousands of feet. Using this and $D = 2R$ in Equation (17), we get

$$\text{increase in thread force} = \pi.D^2(0.5A)/4 = 0.39A.D^2 \qquad (19)$$

As an example, if a bottle with a 3-inch diameter cap is transported to 14,000 feet above the location where it was capped, then $A = 14$, $D = 3$, and the increase in the thread force is,

$$0.39x14x9 = 49 \text{ lbs} \qquad (20)$$

Since this is the same cap as in the example in situation A, the thread force would increase to $63 + 49 = 112$ lbs, even though the sealing force remains unchanged. This could distort the cap and possibly cause the cap threads to jump over the bottle threads.

### 3.1.3 **Situation C**: Add vibration while at altitude

In theory, the worst-case scenario would be when the bottle is tipped upside down, so that the liquid (or powder) is always present and ready to leak out, and the weight of the contents acts like a live load. The equivalent weight of the live load is $W(1+G)$, where $W$ is the dead (static) weight of the bottle and contents and $G$ is the instantaneous acceleration of the bottle expressed in $g$'s. The acceleration of the floor of the truck trailer is known to be about 0.5 $g$'s on average (Singh, et al., 1992), and can get as high as 20 $g$'s when the truck goes over bumps, railroad tracks, and pot holes (Marcondes, et al., 1990). Vibration further complicates the force situation on the bottle by allowing the upside down bottle to tilt slightly off vertical as it vibrates. This has the effect of concentrating the live load at a point as shown in Figure 8 below.

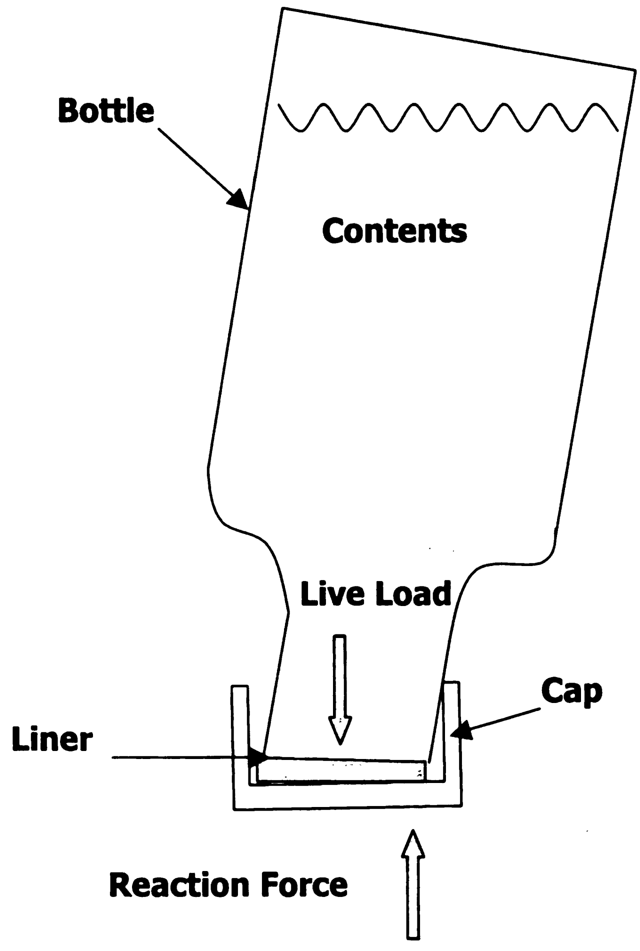



**Figure 8: Free Body Diagram of Bottle and Closure at Altitude with Vibration**

This has the effect of squeezing the liner more on one side than the other. The extra amount of compression depends on how large the live load is, and how long it lasts. Truck trailers typically vibrate up and down on the order of five cycles per second (Pierce, et al., 1992). Assuming an average $G$ of 0.5 during vibration, the live load could go from $W(1+0.5)$ to $W(1-0.5)$ in half a cycle of vibration, or 0.1 seconds.

No matter how large the live load is, or how long it lasts, the net effect of vibration is to *compress and then uncompress* the liner in *rapid* succession. This can easily render the seal force temporarily *zero* at isolated locations. A *rapid* removal of the compression force, such as occurs naturally during vibration, does not allow the liner to recover in time. It takes several seconds, even minutes, for the liner to spring back to its original thickness, once the cap is removed, if it even fully springs back at all. But once the live load is removed, the cap springs back *immediately*. So all during the time that the cap has sprung back, the liner is recovering, and there is a gap between the two. The size of the gap depends on the specifics of the package. Regardless, however, it represents an opportunity for a leak.

### 3.1.4 Observations

Based on this analysis it is clear that both simultaneous vibration and low pressure at high altitudes reduces the overall seal force, which can compromise the closure integrity. The existing test procedures are performed sequentially with each environmental hazard (drop, vibration, pressure, compression) tested once. However in actual shipments in aircraft, vibration and low pressure occur simultaneously. It is therefore expected that the packages tested using the hydrostatic pressure will have an extra seal force, preventing contents from leaking, as opposed to packages tested using the vacuum test which are likely to leak.

The following conclusions can be drawn from the theory discussed in Chapter 3:

- The shippers of these HazMat packages do appear to be following the industry rule regarding the application torque.
- The industry rule is equivalent to requiring that the seal force be the same for all bottles, regardless of cap diameter, and this has the consequence of compressing the liner less for larger caps, so larger caps have greater potential for leaks.
- An increase in altitude affects seal force very little, but raises thread contact forces significantly. This could cause distortion of the bottle neck and cap to the point where the threads begin to jump over each other.
- An increase in altitude affects larger caps much more than smaller ones because the pressure differential acts over a greater area. The potential for leaks is greater for larger caps.
- The effect of vibration is to subject the liner to *intermittent* compression loads. If the liner material is slow to recover, and most are, then vibration produces intermittent gaps which open and close at concentrated pressure points, in step with whatever frequency the bottle vibrates at during transportation.

# 4.0 MATERIALS AND METHODS

Several types of commercially available, UN certified hazardous material combination packages were obtained from three US manufacturers in consultation with FAA. These packages represented the types, which were known to have shown package incident failures in air cargo shipments. Several test methods were developed to evaluate the performance of these types of packages. The test methods represent the various types of conditions that these types of packages are likely to observe in high altitude shipments. This chapter discusses the different UN certified packages that were tested, the various types of test equipment used, and the test methods.

## 4.1 Package Types Tested

The UN certified packages were obtained from three different US manufacturers. These were:

1. LABELMASTER Inc., Chicago, IL (www.labelmaster.com)
2. CARGOpak Corporation, Raleigh, NC (www.cargopak.com)
3. HAZMATPAC Inc., Houston, TX (www.hazmatpac.com)

Each of the various combination packages obtained and tested in this study is discussed in the next section. The descriptions provided in the following section is information provided by the HazMat package supplier.

### 4.1.1 LABELMASTER Packages

**HMS-08**

Consists of a PVC plasticoated glass bottle and a plastic lid with a flat liner. The lid is sealed with PP tape and placed inside a PP bag, which is sealed with a nylon tie. The bag is wrapped in a vermiculite pad and placed inside a steel canister. This canister is then placed inside a double-wall fiberboard insert. The whole setup is contained inside another PP bag and sealed with a nylon tie before being boxed and sealed inside a double-wall fiberboard carton.

**UN950PPT**

Consists of a PE bottle and cap (with flat liner), which is sealed with PP tape and placed in a fiberboard insert. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

**UN950GPT**

Consists of glass bottle and a plastic cap with a flat liner, which is sealed with PP tape and then placed in a XEBEC pouch with zip lock. The setup is then placed in a fiberboard box and sealed

**UN16FFPS**

Consists of a glass bottle and plastic cap with a PE cone liner. The bottle is sealed with PP tape and placed inside two PS end caps at the top and bottom. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

**UN32FFPS**

Consists of a glass bottle and plastic cap with a PE cone liner. The bottle is sealed with PP tape and placed inside an enclosure made of two molded PS pieces. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

**UNHWS16**

Consists of a wide mouth glass bottle and plastic cap with a flat liner. The bottle is sealed with PP tape and placed inside two PS end caps at the top and bottom and a central PS body piece. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

**UN32NPVB**

Consists of wide mouth Nalgene PE bottle and a liner less plastic cap, which is sealed with PP tape and then placed in a XEBEC pouch with zip lock. The setup is then placed in a fiberboard box and sealed.

**HMSP-32N**

Consists of four Nalgene PE bottle with liner less plastic caps. These are completely enclosed in a three-piece (two end pieces and a center-body piece) PS setup. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a double-wall fiberboard box and sealed.

**UN32PPS**

Consists of a PE bottle and a cap with a cone liner, which is sealed with PP tape and placed in a fiberboard insert. A PS end piece is placed on the top end of the bottle. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

**UN4FFPS**

Consists of a glass bottle and plastic cap with a PE cone liner. The bottle is sealed with PP tape and placed inside two PS end caps at the top and bottom and a central PS body piece. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

**UAC32FPS**

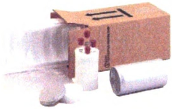
Consists of a glass bottle and plastic cap (with PE cone liner). The bottle is sealed with PP tape and placed in a double-wall fiberboard insert and between two double-wall fiberboard end cushions. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a double-wall fiberboard box and sealed.

**UN32FAPS**

Consists of a glass bottle and plastic cap with a PE cone liner. The bottle is sealed with PP tape and placed inside an enclosure made of two molded PS pieces. This setup after being placed inside a PP bag and closed with a nylon tie is then placed inside a fiberboard box and sealed.

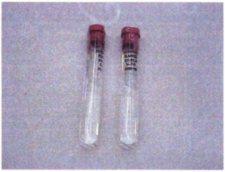
**HINF630**

Consists of four 10ml drawtubes, which have friction rubber closures. These are placed in a preformed cushioned encasing with a cushion top. The cushion is then placed within an aluminum canister with a screw on aluminum lid. This setup after being sealed in a zip lock PP bag is placed inside an enclosure made of two-piece molded PS cushion and then put in a fiberboard box and sealed.



**PACK 1**

Glass test tubes with rubber closure held in place by adhesive tape. These are not DOT approved HazMat packages but as a common practice are used by clinics to ship human and animal blood and other specimens to test using the single parcel carriers.



**PACK 2**

Glass test tubes with rubber closure (no adhesive tape). These are not DOT approved HazMat packages but as a common practice are used by clinics to ship human and animal blood and other specimens to test using the single parcel carriers.

### 4.1.2 HAZMATPAC, Inc. Packages

**UNE151**

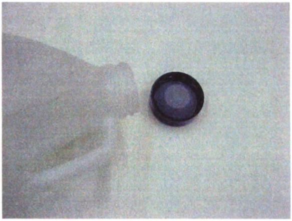

8oz. Cap is teflon lined

**UN112**

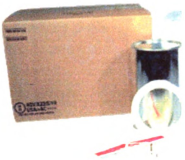
**HAZMATPAC**'s 4GV United Nations certified packaging system provides all of the required components for the safe transport of hazardous materials by air, ground and water. The 4GV series passes **ISTA** International Safe Transit associations Project 3 testing for the overnight environment and meets Project 1A testing. Each packaging system comes completely assembled with easy to read instructions for effortless final packaging. All **HAZMATPAC** United Nations certified packaging systems have been third party tested by **WYLE** Laboratories to ensure unbiased test results. It is the responsibility of the person offering a hazardous material for transportation to ensure that such packagings are compatible with their lading. Stock numbers are for complete packaging systems.

**4GV/X14/S/02/USA/+AC1604**

1/2 gallon plastic



**UN1541**



**HAZMATPAC**'s 4GV United Nations certified packaging system provides all of the required components for the safe transport of hazardous materials by air, ground and water. The 4GV series passes **ISTA** International Safe Transit Associations Project 3 testing for the overnight environment and meets Project 1A testing. This certification includes testing requirements of 49 CFR section 173.226 for Materials Toxic by Inhalation Division 6.1 Packaging Group I, Hazard Zone A. Each packaging system comes completely assembled with easy to read instructions for effortless final packaging. All **HAZMATPAC** United Nations certified packaging systems have been third party tested by **WYLE** Laboratories to ensure unbiased test results. It is the responsibility of the person offering a hazardous material for transportation to ensure that such packagings are compatible with their lading. Stock numbers are for complete packaging systems.

**4GV/X23/S/02/USA/+AC1609**

Caps are teflon lined

**UN61, UN 62**

**HAZMATPAC**'s 4GV United Nations certified packaging system provides all of the required components for the safe transport of hazardous materials by air, ground and water. The 4GV series passes **ISTA** International Safe Transit Associations Project 3 testing for the overnight environment and meets Project 1A testing. Each packaging system comes completely assembled with easy to read instructions for effortless final packaging. All **HAZMATPAC** United Nations certified packaging systems have been third party tested by **WYLE** Laboratories to ensure unbiased test results. It is the responsibility of the person offering a hazardous material for transportation to ensure that such packagings are compatible with their lading. Stock numbers are for complete packaging systems.

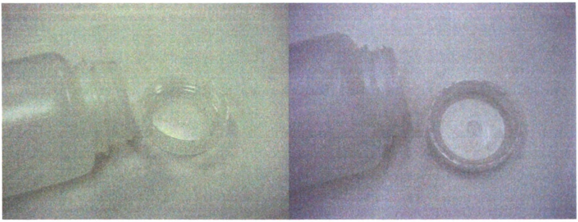
**4GV/X4/S/02/USA/+AC1603**

HAZMATPAC's new universal absorbent lined bagging system is designed to provide sufficient absorbent material around the container while achieving the necessary cushioning to pass the 4GV tests requirements. The bag lining system includes a proprietary design of universal polypropylene absorbent folded in a unique way to provide the most layers of absorbent at the top and the bottom of the bag, where it is needed most. The bag lining system is the cleanest and easiest form of universal absorbent available. Packaging of each bottle is accomplished with three easy steps:

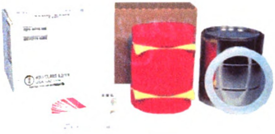
1. Partially pull out the absorbent lining and insert the container.
2. Hold on to the outer bag and let the weight of the container push the absorbent lining with the container to the bottom of the bag.
3. Fold the top flaps of the absorbent lining over the top of the container and close with a twist tie.

The bag lining system provides a universal, clean and easy way to package your container in the safest 4GV United Nations Certified packaging system.
Tare weight for UN-57 is only 1.46 pounds

**Wide mouth natural HDPE**



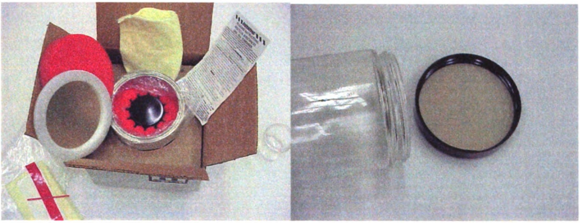
**UNIS80**



HAZMATPAC's Infectious Substance shipping container is designed to safely transport Class 6.2 substances worldwide. The complete packaging system safely ships one 8oz.straight sided jar or up to twelve 10ML inner receptacles. HAZMATPAC's Infectious Substance shipping container is tested to meet or exceed all of the current regulations for Infectious Substances. The new regulations include the "4GU" standard which is very similar to the "V" standard, allowing for inner receptacles of any type to be assembled within an intermediate (secondary) packaging. The complete packaging system includes all of the required components for the safe transport of infectious substances by air, ground and water.

4GU/CLASS 6.2/02/USA/+AC****

**UN-IS80 ONE - 8oz. STRAIGHT SIDED INFECTIOUS SUBSTANCE SHIPPER**

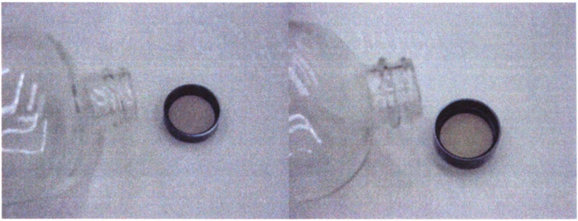


## UN51, UN52

**HAZMATPAC**'s 4GV United Nations certified packaging system provides all of the required components for the safe transport of hazardous materials by air, ground and water. The 4GV series passes **ISTA** International Safe Transit Associations Project 3 testing for the overnight environment and meets Project 1A testing. Each packaging system comes completely assembled with easy to read instructions for effortless final packaging. All **HAZMATPAC** United Nations certified packaging systems have been third party tested by **WYLE** Laboratories to ensure unbiased test results. It is the responsibility of the person offering a hazardous material for transportation to ensure that such packagings are compatible with their lading. Stock numbers are for complete packaging systems.

**4GV/X4/S/02/USA/+AC1603**

Caps are teflon lined
16 oz. UN-51, 32 oz. UN-52



## UN78, UN79

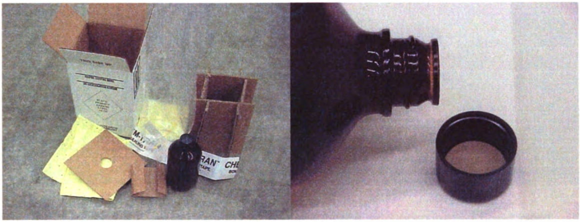
**HAZMATPAC**'s 4GV United Nations certified packaging system provides all of the required components for the safe transport of hazardous materials by air, ground and water. The 4GV series passes **ISTA** International Safe Transit Associations Project 3 testing for the overnight environment and meets Project 1A testing. Each packaging system comes completely assembled with easy to read instructions for effortless final packaging. All **HAZMATPAC** United Nations certified packaging systems have been third party tested by **WYLE** Laboratories to ensure unbiased test results. It is the responsibility of the person offering a hazardous material for transportation to ensure that such packagings are compatible with their lading. Stock numbers are for complete packaging systems.

**4GV/X4/S/02/USA/+AC1603**

Caps are teflon lined    UN-78: 16oz., UN-79: 32 oz.

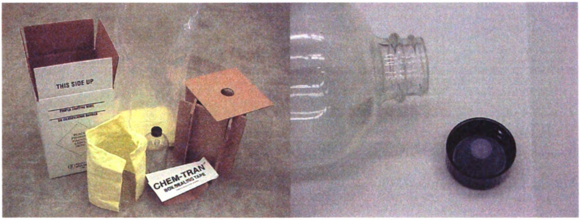
### 4.1.3 CARGOpak Corp. Packages

**V1-1000N Glass Pax**



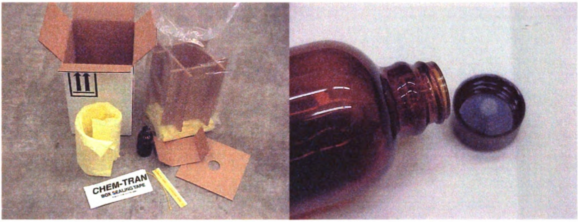
This combination package consists of a glass bottle and corrugated fiberboard shipper shown above.

**V1-0500N Glass Pax**



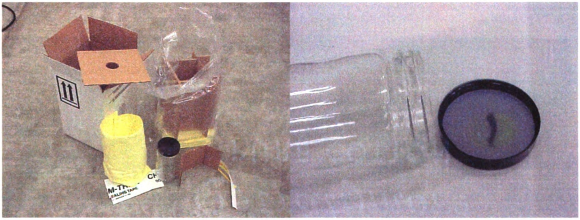

This combination package consists of a glass bottle and corrugated fiberboard shipper shown above.

**V1-0125-N Glass Pax**



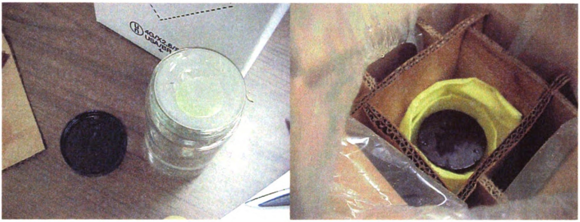

This combination package consists of a glass bottle and corrugated fiberboard shipper shown above.

**V1-0500W Glass Pax**



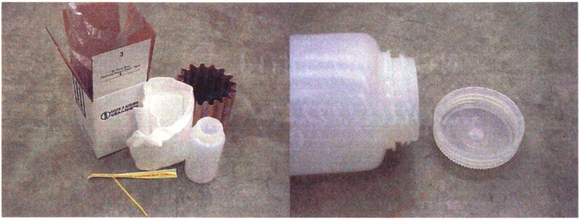
This combination package consists of a wide-mouth glass bottle and corrugated fiberboard shipper shown above.

**CT-SP-0002**



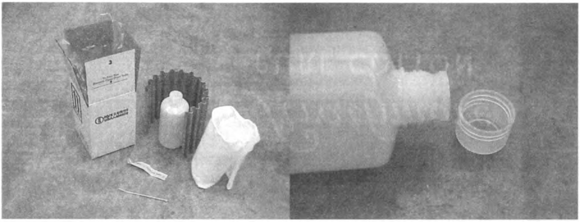
This combination package consists of a wide-mouth glass bottle and corrugated fiberboard shipper shown above.

**CT-1-92-1000-W**



This combination package consists of a plastic bottle and corrugated fiber board shipper shown above.

**CT-1-92-1000-N**



This combination package consists of a plastic bottle and corrugated fiberboard shipper shown above.

## 4.2 TEST EQUIPMENT:

In order to conduct simultaneous low pressure and vibration on the test packages the following equipment was used:

### 4.2.1 Electro-hydraulic Vibration Table

A Lansmont electro-hydraulic vibration table (Model 7000) was used. The vibration table controller was capable of being programmed to perform sinusoidal or random vibration tests. For this study random vibration tests were conducted. The Power Density Spectrums used for vibration simulation were for Truck/Air combination shipments or Truck Only shipments. These spectrums were based on the recommended vibration levels for these modes of transport as shown in ASTM D4728 and ASTM D4169, Assurance Level II.

Figures 9 and 10, below show the vibration test profiles used for this study. Figure 9 is the "Truck Assurance Level II Random Vibration Test Profile" used for testing in Phase V and Figure 10 depicts the "Truck/Air Transport Vibration Test Profile" that was used during the testing of Phases I, II, and IV.

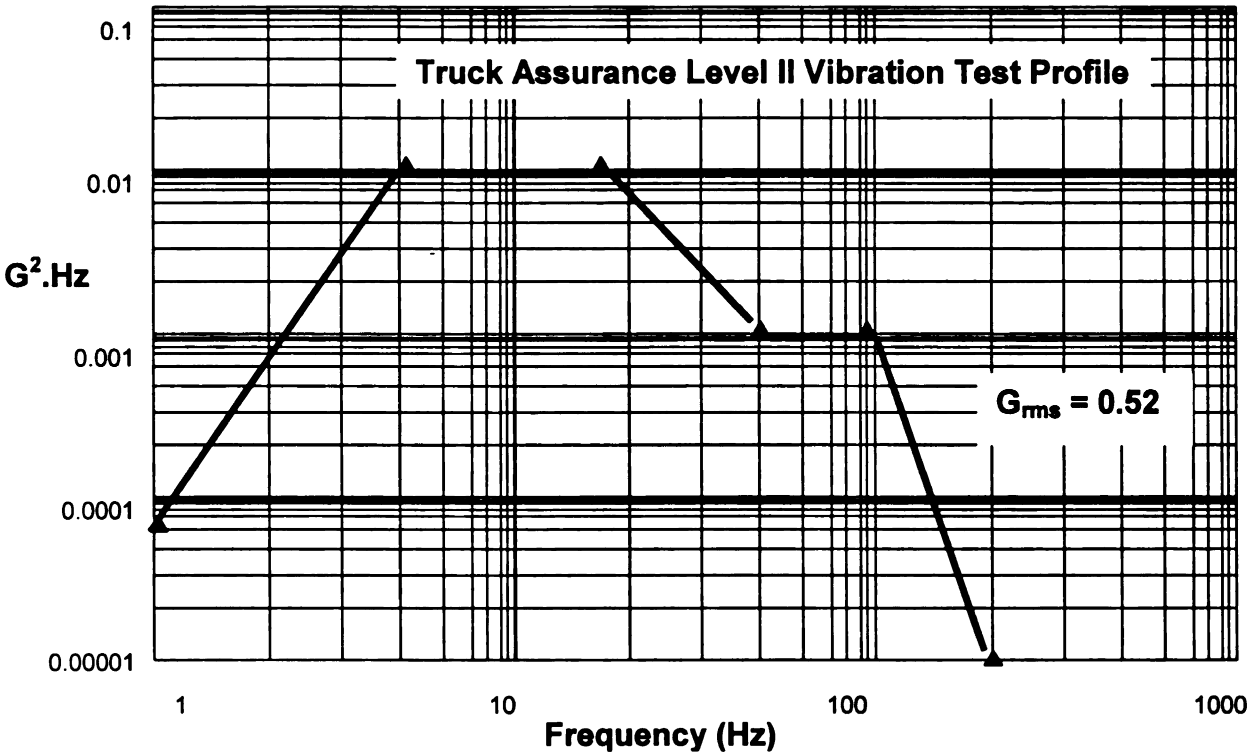



**Figure 9: Truck Assurance Level II Random Vibration Test Profile**

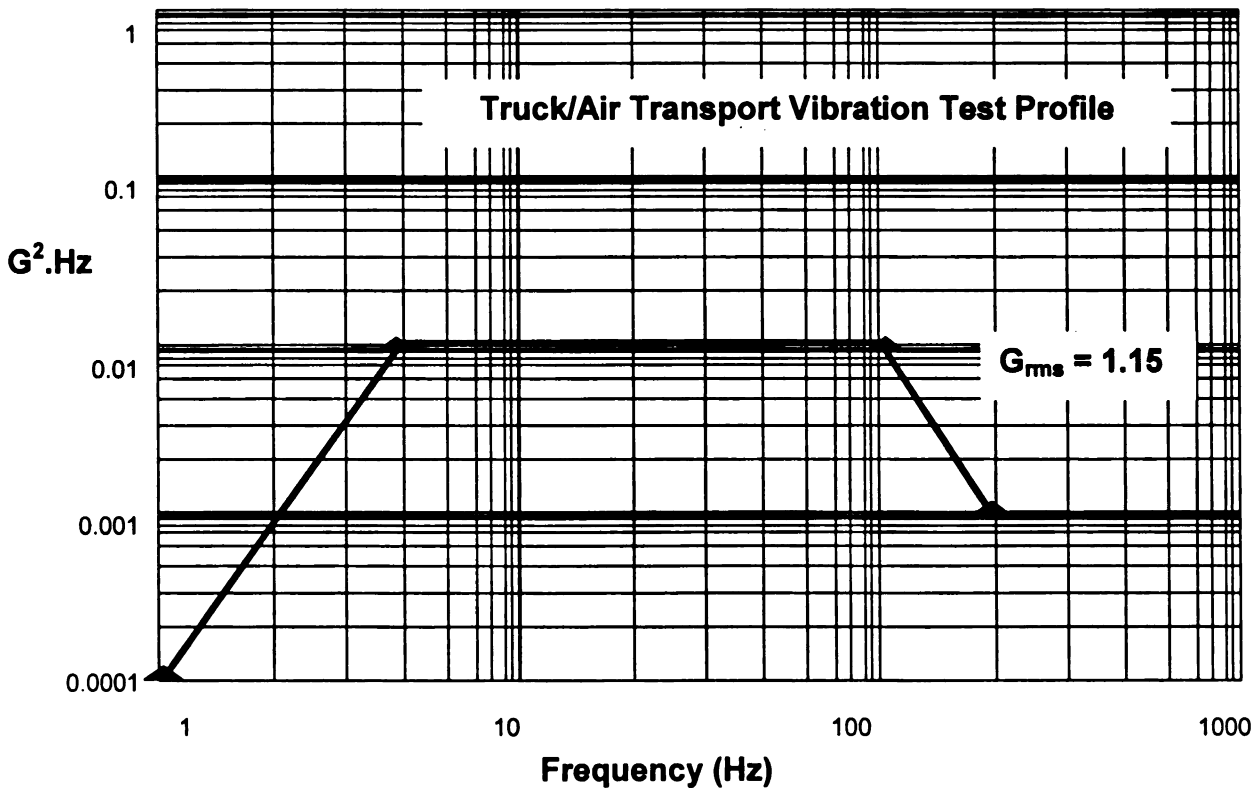



**Figure 10: Truck/Air Transport Vibration Test Profile**

### 4.2.2 Vacuum Chamber

A Tek-Vac Industries Inc. Vacuum Chamber System (Model VC-S222-SE) was used for this study. This system was capable of achieving an altitude of up to 50,000 feet with an accuracy of ± 100 feet. The system allowed a maximum size package of eight cubic feet (2' x 2' x 2') to be tested in the chamber. The pressure gauge was capable of a maximum vacuum readings of 30 inHg (101.6 kPa). The pressure drop based on altitude was determined from NACA Report 538(1936).

Figure 11 shows the test setup to conduct simultaneous low pressure and vibration tests on combination packages.

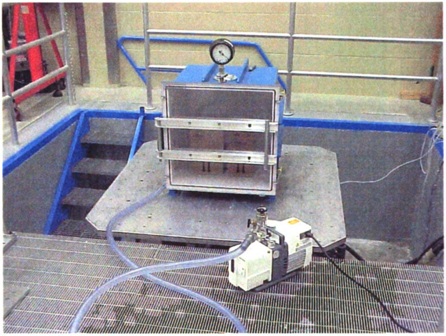


**Figure 11: Experimental Setup with Test Packages**

In addition to the above test equipment a torque tester was used to measure both application and removal torque on certain types of packages.

### 4.2.3 Closure Torque Tester

A SecurePak Digital Model Torque Tester was used to measure application and removal torque levels on closures for certain packages. Figure 12 shows the torque tester used. (Calibration performed according to ASTM D-3474 by SecurePak on 11/27/2001)

**Figure 12: Torque Tester**

## 4.3 Test Methods

This study was conducted over five test phases. Each test phase represents the different conditions of low pressure and vibration that packages are likely to be exposed during high altitude shipments. Based on the results of the preliminary study presented by Singh and Burgess (2000), a new test method was proposed to ASTM for low pressure testing of packages that undergo high altitude shipments.

In addition to the above findings, United Parcel Service presented a study to ASTM describing the altitude, temperature, and duration that packages undergo in the single parcel shipping environment (ASTM, 2001). The study showed the following key observations (ASTM D6653-01):

- Cargo air jets typically are pressurized to approximately 2,438 m (8,000 ft). Temperature is maintained to approximately 20 to 23 °C (68 to 74 °F)
- Packages transported on ground may experience altitudes as high as 3,658 m (12,000 ft) when shipped over certain mountain passes especially in Colorado. Temperature extremes range from –15 to 30 °C (5 to 86 °F) with average mean temperatures of approximately –4 to 18 °C (25 to 64 °F)
- Non-pressurized feeder aircraft typically fly at approximately 3,963 m to 4,877 m (13,000 to 16,000 ft). the highest recorded altitude in a non-pressurized feeder aircraft was 6,017 m (19,740 ft). Temperature recordings ranged from approximately –4 to 24 °C (25 to 75 °F)

Based on the above recommendations, ASTM developed and approved a new test method, D6653-01, in 2001 titled, "Standard Test Methods for Determining the Effects of High Altitude on Packaging Systems by Vacuum

Method" (ASTM, 2002). The test method recommends the procedure to apply vacuum to packages that undergo ground or air shipments at high altitude.

ASTM D 4169 describes three different test level intensities (Assurance Levels I, II and III) for evaluating shipping container performance. These test intensity levels are related to uncertainties in environmental conditions. Assurance Level I claims a high level of intensity, but a low probability of occurring in transport environments. This leads many people to consider Assurance Level I as conservative, with plenty of safety factor built in. Upon consultation with FAA and DOT, and from past experiences of various tests conducted for the Consortium of Distribution Packaging, a decision was made to go use Assurance Level II for this project. Assurance level II is also the most commonly and regularly used intensity level by the testing facilities. The selection of this assurance level does not limit or restrain a testing facility to perform the proposed procedure at a higher level (Assurance Level I). A minimum assurance level of II must, however, be used.

The five test phases conduced are discussed in detail in this section.

### 4.3.1 Phase I (Truck/Air Simulation, 14,000 Feet Pressure Differential)

This test phase consisted of evaluation of UN approved HazMat packages from the three US suppliers mentioned above. The test consisted of simultaneous low pressure and vibration representing an altitude of 14,000 feet and a combined truck/air vibration for mode of shipment.

*Test Procedure:*

- Packages were conditioned at 73.4 ± 3.6°F for a minimum of 24 hours before testing
- The primary containers were filled to the fill-level recommended and proper closure torque was applied
- Secondary packaging was applied, as if preparing for shipping, in accordance with the manufacturer's instructions
- Two samples of each kind of SKU were used for this phase
- The test specimen was placed in the top-down position in the vacuum chamber and the vacuum chamber was placed on an electro-hydraulic vibration table
- After sealing the vacuum chamber shut, the vacuum source was turned on and adjusted to a rate of 305 meters in 30-60 seconds as recommended in ASTM D6653-01. This replicates take off conditions on an airplane of 1000 – 2000 feet/minute.
- A vacuum of 59.5 kPa (pressure equivalent of 14,000 feet) was achieved with a permissible error margin of ±2%

- While maintaining the vacuum of 59.5 kPa, the vibration table was operated for 30 minutes using random mode simulation of a truck/air-shipping environment (Assurance level II, ASTM D 4169) representing shipments of 250 miles.
- The chamber inlet valve was opened and the vacuum released at a rate of 305 meters (1000 feet) per 30-60 seconds
- The chamber cover was removed to retrieve the test specimen
- Any leakage observed was recorded
- Figure 5 shows a description of the test setup. The samples were placed in the vacuum chamber and the chamber was placed on an electro-hydraulic vibration table.

### 4.3.2 Phase II (Truck/Air Simulation Vibration)

This test phase consisted of evaluation of UN approved HazMat packages obtained from Labelmaster Inc. The test consisted of only vibration simulation representing a truck/air mode of shipment.

*Test Procedure:*

- Packages were conditioned at 73.4 ± 3.6°F for a minimum of 24 hours before testing
- The primary containers were filled to the fill-level recommended and proper application torque was applied and recorded

- A vertical mark was applied at the meeting point of the container and closure to monitor for closure back offs after a pressure differential application
- Two samples of each kind of SKU were used for this phase
- Secondary packaging was applied, as if preparing for shipping, in accordance with the manufacturer's instructions
- The test specimen was placed in the top-down position on the vibration table
- The vibration table was operated for 30 minutes using random mode simulation of a truck/air shipping environment (Assurance level II, ASTM D 4169)
- The samples were examined after test for any leakage, closure back offs and removal torques

### 4.3.3 Phase III (14,000 Feet, Vacuum Only)

This test phase consisted of evaluation of UN approved HazMat packages obtained from Labelmaster Inc. The test consisted of only low pressure simulation representing a 14,000 ft high altitude shipment.

*Test Procedure:*

- Packages were conditioned at 73.4 ± 3.6°F for a minimum of 24 hours before testing

- The primary containers were filled to the recommended fill-level and proper application torque was applied and recorded
- A vertical mark was applied at the meeting point of the container and closure to monitor for closure back offs after a pressure differential application
- Two samples of each kind of SKU were used for this phase
- Secondary packaging was applied, as if preparing for shipping, in accordance with the manufacturer's instructions
- The test specimens were placed in the top-down position in the vacuum chamber
- After sealing the vacuum chamber shut, the chamber inlet valve was closed and the outlet valve opened
- The vacuum source was turned on and adjusted to a rate of 305 meters (1000 feet) per 30-60 seconds
- A vacuum of 59.5 kPa (pressure equivalent of 14,000 feet) was achieved with a permissible error margin of ±2%
- The two identical samples were subjected to this vacuum of 59.5 kPa for 30 minutes
- The chamber inlet valve was partially opened and the vacuum released at a rate of 305 meters (1000 feet) per 30-60 seconds
- The chamber cover was removed to retrieve the test specimen
- Any leakage, closure back offs and the removal torques of the samples were recorded

### 4.3.4 Phase IV (Truck/Air Simulation, 8,000 Feet Pressure Differential)

This test phase consisted of evaluation of UN approved HazMat packages from all three US suppliers mentioned above. The test consisted of simultaneous low pressure and vibration representing an altitude of 8,000 feet and a combined truck/air vibration for mode of shipment. This represented shipments in commercial and cargo pressurized aircraft.

*Test Procedure:*

- Packages were conditioned at 73.4 ± 3.6°F for a minimum of 24 hours before testing
- The primary containers were filled to the fill-level recommended and proper closure torque was applied
- Secondary packaging was applied, as if preparing for shipping, in accordance with the manufacturer's instructions
- Two samples of each kind of SKU were used for this phase
- The test specimen was placed in the side-ways position in the vacuum chamber and the vacuum chamber was placed on an electro-hydraulic vibration table
- After sealing the vacuum chamber shut, the vacuum source was turned on and adjusted to a rate of 305 meters (1000 feet) per 30-60 seconds as recommended in ASTM D6653-01
- A vacuum of 75.3 kPa (pressure equivalent of 8,000 feet) was achieved with a permissible error margin of ±2%

- While maintaining the vacuum of 75.3 kPa, the vibration table was operated for 3 hours using random mode simulation of a truck/air-shipping environment (Assurance level II, ASTM D 4169).
- The chamber inlet valve was opened and the vacuum released at a rate of 305 meters (1000 feet) per 30-60 seconds
- The chamber cover was removed to retrieve the test specimen
- Any leakage observed was recorded

### 4.3.5 Phase V (Truck Simulation, 8,000 Feet Pressure Differential)

This test phase consisted of evaluation of UN approved HazMat packages from the three US suppliers mentioned above. The test consisted of simultaneous low pressure and vibration representing an altitude of 8,000 feet and a truck only random vibration for mode of shipment. This represents ground shipments in high altitude passes.

*Test Procedure:*

- Packages were conditioned at 73.4 ± 3.6°F for a minimum of 24 hours before testing
- The primary containers were filled to the fill-level recommended and proper closure torque was applied
- Secondary packaging was applied, as if preparing for shipping, in accordance with the manufacturer's instructions
- Two samples of each kind of SKU were used for this phase

- The test specimen was placed in the top-down position in the vacuum chamber and the vacuum chamber was placed on an electro-hydraulic vibration table
- After sealing the vacuum chamber shut, the vacuum source was turned on and adjusted to a rate of 305 meters (1000 feet) per 30-60 seconds as recommended in ASTM D6653-01
- A vacuum of 75.3 kPa (pressure equivalent of 8,000 feet) was achieved with a permissible error margin of ±2%
- While maintaining the vacuum of 75.3 kPa, the vibration table was operated for 3 hours using random mode simulation of a truck-shipping environment (Assurance level II, ASTM D 4169).
- The chamber inlet valve was opened and the vacuum released at a rate of 305 meters (1000 feet) per 30-60 seconds
- The chamber cover was removed to retrieve the test specimen
- Any leakage observed was recorded

On completion of the above tests, all data was recorded. The results from the above tests are discussed in the next chapter.

## 5.0 DATA AND RESULTS

This chapter discusses the data and results from the five test phases conducted in this study. The various pictures describing packages that leaked and methods to monitor cap back-off are shown at the end of this chapter.

### 5.1 **Phase I** (Truck/Air Simulation, 14,000 Feet Vacuum)

Table 7 describes the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase I procedure described in 4.3.1. These packages were obtained from Labelmaster Inc. The rows showing shaded regions represent containers that leaked.

Table 8 describes the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase I procedure obtained from CARGOpak Corp. Table 9 describes the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase I procedure obtained from HAZMATPAC Inc.

The results from these tests showed that there were a large percentage of packages that are currently approved for both the vibration and pressure differential tests in accordance with existing DOT requirements that showed leaks when simultaneously tested for partial vacuum and vibration representing an un-pressurized air shipment at 14,000 ft. It is also interesting to note that all the screw top closures evaluated had removal torque levels, but could not maintain package integrity.

**Table 7:** Phase I - Test Results for Labelmaster Inc.
(Truck/Air Simulation, 14,000 Feet Vacuum)

| SKU | *PHASE I* | | | |
|---|---|---|---|---|
| | SAMPLE A | | SAMPLE B | |
| | AT | RT | AT | RT |
| HMS-08 | 21.2 | 19.3 | 21.6 | 16.5 |
| UN950PPT | 20.1 | 15.5 | 20.0 | 16.4 |
| UN950GPT | 11.2 | 11.0 | 11.1 | 8.1 |
| UN16FFPS | 11.2 | 10.0 | 11.1 | 8.8 |
| UN32FFPS | 16.3 | 12.8 | 16.3 | 12.9 |
| UNHWS16 | 35.2 | 31.5 | 35.1 | 22.1 |
| UN32NPVB | 56.0 | 35.7 | 56.4 | 32.6 |
| HMSP-32N | 18.1 | 13.4 | 18.2 | 14.6 |
| UN32PPS | 20.1 | 19.6 | 20.1 | 19.1 |
| UN4FFPS | 11.1 | 9.9 | 11.3 | 9.2 |
| UAC32FPS | 16.0 | 15.8 | 16.1 | 14.2 |
| UN32FAPS | 11.2 | 9.2 | 11.3 | 7.5 |
| PACK 1 | | | | |
| PACK 2 | | | | |

*The shaded cells represent containers that leaked

**Table 8:** Phase I – Test Results for HAZMATPAC, Inc.
(Truck/Air Simulation, 14,000 Feet Pressure Differential)

| SKU | *PHASE I* | | | |
|---|---|---|---|---|
| | SAMPLE A | | SAMPLE B | |
| | AT | RT | AT | RT |
| UNE151 | 11.1 | 7.2 | 11.1 | 7.3 |
| UN112 | 20.1 | 18.3 | 20.3 | 18.8 |
| UN1541 | 11.4 | 6.5 | 11.2 | 7.1 |
| UN61 | 20.1 | 17.4 | 20.1 | 18.5 |
| UN 62 | 56.1 | 47.1 | 56.6 | 44.9 |
| UNIS80 | 35.4 | 29.7 | 35.6 | 32.3 |
| UN51 | 11.3 | 8.4 | 11.2 | 6.0 |
| UN52 | 16.1 | 15.1 | 16.1 | 13.4 |
| UN78 | 35.1 | 30.8 | 35.1 | 32.1 |
| UN79 | 35.1 | 31.8 | 35.3 | 32.8 |

**Table 9:** Phase I: Test Results for CARGOpak, Corp.
(Truck/Air Simulation, 14,000 Feet Vacuum)

| SKU | *PHASE I* | | | |
|---|---|---|---|---|
| | SAMPLE A | | SAMPLE B | |
| | AT | RT | AT | RT |
| CT-SP-0002 | 21.2 | 19.2 | 21.2 | 19.8 |
| CT-1-92-1000-N | 33.4 | 20.5 | 33.6 | 21.6 |
| CT-1-92-1000-W | 56.2 | 32.3 | 57.1 | 36.7 |
| CT-4-92-1000-N | 33.1 | 18.1 | 33.2 | 16.7 |
| V1-0125-N | 11.5 | 9.9 | 11.1 | 9.2 |
| V1-0500N | 11.4 | 9.8 | 11.1 | 9.2 |
| V1-1000N | 20.4 | 10.5 | 20.1 | 14.6 |
| V1-0500W | 35.2 | 28.2 | 35.3 | 22.8 |

*The shaded cells represent containers that leaked

## 5.2 **Phase II** (Truck/Air Simulation Vibration)

Table 10 shows the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase II procedure described in 4.3.2. These packages were obtained from Labelmaster Inc. The rows showing shaded regions represent containers that leaked. The data shows that only one container leaked as a result of performing the tests with vibration.

**Table 10:** Phase II – Test Results for Lablemaster Inc.
(TRUCK/AIR SIMULATION VIBRATION)

| SKU | PHASE II | | | |
|---|---|---|---|---|
| | SAMPLE A | | SAMPLE B | |
| | AT | RT | AT | RT |
| HMS-08 | 21.1 | 19.8 | 21.2 | 19.5 |
| UN950PPT | 20.5 | 18.2 | 20.5 | 18.9 |
| UN950GPT | 11.3 | 10.8 | 11.2 | 10.2 |
| UN16FFPS | 11.1 | 9.8 | 11.1 | 9.5 |
| UN32FFPS | 16.1 | 13.8 | 16.0 | 13.5 |
| UNHWS16 | 35.3 | 27.7 | 35.1 | 31.1 |
| UN32NPVB | 56.7 | 44.6 | 56.3 | 47.7 |
| HMSP-32N | 18.3 | 16.0 | 18.3 | 14.6 |
| UN32PPS | 20.0 | 17.7 | 20.3 | 17.7 |
| UN4FFPS | 11.1 | 10.3 | 11.5 | 10.1 |
| UAC32FPS | 16.0 | 13.2 | 16.1 | 13.7 |
| UN32FAPS | 11.1 | 10.2 | 11.2 | 9.8 |
| PACK 1 | | | | |
| PACK 2 | | | | |

*The shaded cells represent containers that leaked

**5.2 Phase III** (14,000 feet, vacuum only)

Table 11 shows the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase III procedure described in 4.3.3. These packages were also obtained from Labelmaster Inc. The data shows no containers leaked.

**Table 11:** Phase III – Test Results for Labelmaster Inc.
(14,000 FEET – VACUUM)

| SKU | *PHASE III* | | | |
|---|---|---|---|---|
| | SAMPLE A | | SAMPLE B | |
| | AT | RT | AT | RT |
| HMS-08 | 21.2 | 18.3 | 21.2 | 17.0 |
| UN950PPT | 20.0 | 17.8 | 20.1 | 17.2 |
| UN950GPT | 11.1 | 10.2 | 11.2 | 10.7 |
| UN16FFPS | 11.1 | 8.8 | 11.1 | 9.2 |
| UN32FFPS | 16.3 | 14.2 | 16.0 | 14.8 |
| UNHWS16 | 35.5 | 28.7 | 35.5 | 32.0 |
| UN32NPVB | 56.1 | 40.3 | 56.3 | 46.1 |
| HMSP-32N | 18.1 | 14.1 | 18.0 | 13.6 |
| UN32PPS | 20.2 | 17.7 | 20.1 | 17.7 |
| UN4FFPS | 11.1 | 10.9 | 11.2 | 11.0 |
| UAC32FPS | 16.0 | 15.8 | 16.1 | 15.2 |
| UN32FAPS | 11.1 | 10.8 | 11.1 | 10.9 |
| PACK 1 | | | | |
| PACK 2 | | | | |

Based on the results seen in Phase I, II, and III, it is clear that simultaneous testing of low pressure and vibration produces the types of leaks representative in real life observations made by FAA. Testing packages sequentially for low pressure and vibration alone shows an extremely small number of package failures. The current DOT specification for pressure differential test requires packages to have met the 95 kPa requirement. While this may have been accomplished using the hydrostatic pressure test, it is clear from the results of Phase I, II, and III, that the packages that showed leaks in Tables 7-9 were tested at 59.5 kPa. It is evident that a vacuum level representing 95 kPa, would likely increase the number of leaks for these packages.

**5.4 Phase IV** (Truck/Air Simulation, 8,000 Feet Pressure Differential)

Table 12 shows the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase IV procedure described in 4.3.4. These tests represent conditions that packages would undergo when traveling in pressurized commercial and cargo aircraft. These aircraft are pressurized to represent 8000 ft altitude conditions (75.3 kPa vacuum requirement). The vibration levels used were a combined truck/air spectrum. These packages were of the same type tested in Phase I, II, and III. The rows showing shaded regions represent containers that leaked.

The results showed that four of the UN approved packages failed this test.

**Table 12:** Phase IV – Test Results for Labelmaster Inc.
(Truck/Air Simulation, 8,000 Feet Pressure Differential)

| SKU | *PHASE IV (TRUCK/AIR)* | | | |
|---|---|---|---|---|
| | **SAMPLE A** | | **SAMPLE B** | |
| | **AT** | **RT** | **AT** | **RT** |
| HMS-08 | 21.2 | 19.6 | 21.6 | 18.5 |
| UN950PPT | 20.1 | 18.6 | 20.3 | 19.8 |
| UN950GPT | 11.2 | 3.6 | 11.1 | 9.2 |
| UN16FFPS | 11.5 | 11.0 | 11.3 | 10.9 |
| UN32FFPS | 16.1 | 15.8 | 16.4 | 15.4 |
| UNHWS16 | 35.2 | 28.2 | 35.6 | 32.0 |
| UN32NPVB | 56.2 | 35.6 | 56.0 | 38.4 |
| HMSP-32N | 18.1 | 13.4 | 18.0 | 12.2 |
| UN32PPS | 20.3 | 19.8 | 20.0 | 16.8 |
| UN4FFPS | 11.1 | 10.9 | 11.2 | 10.3 |
| UAC32FPS | 16.1 | 14.2 | 16.2 | 12.5 |
| UN32FAPS | 11.2 | 10.9 | 11.3 | 10.8 |
| PACK 1 | | | | |
| PACK 2 | | | | |

*The shaded cells represent containers that leaked

## 5.5 Phase V (Truck Simulation, 8,000 Feet Pressure Differential)

Table 13 shows the application and removal torque levels on two sets of UN approved HazMat packages tested in accordance with Phase V procedure described in 4.3.5. These tests represent conditions that packages would undergo, when traveling in trucks at high altitudes. The vibration spectrum used was for a Composite Truck Transport. These packages were of the same type tested in Phase I, II, and III. The rows showing shaded regions represent containers that leaked. The results showed that two of the UN approved packages failed this test.

Based on the results from Phase IV and V it is evident that the existing DOT specifications for ground shipments at high altitude are not adequate to prevent leaks from HazMat packages. Similarly these UN approved packages based on ICAO requirements do not provide adequate integrity when shipped in pressurized cargo aircraft.

**Table 13:** Phase V – Test Results for Labelmaster Inc.
(Truck Simulation, 8,000 Feet Pressure Differential)

| SKU | PHASE V (TRUCK ONLY) | | | |
|---|---|---|---|---|
| | SAMPLE A | | SAMPLE B | |
| | AT | RT | AT | RT |
| HMS-08 | 21.1 | 17.3 | 21.1 | 15.8 |
| UN950PPT | 20.0 | 17.5 | 20.3 | 16.4 |
| UN950GPT | 11.1 | 10.8 | 11.0 | 10.2 |
| UN16FFPS | 11.1 | 10.2 | 11.2 | 10.5 |
| UN32FFPS | 16.0 | 14.8 | 16.1 | 15.2 |
| UNHWS16 | 35.1 | 31.5 | 35.2 | 30.1 |
| UN32NPVB | 56.1 | 38.9 | 56.4 | 40.2 |
| HMSP-32N | 18.0 | 14.3 | 18.3 | 16.8 |
| UN32PPS | 20.3 | 17.2 | 20.4 | 18.8 |
| UN4FFPS | 11.1 | 10.3 | 11.3 | 10.4 |
| UAC32FPS | 16.0 | 14.5 | 16.2 | 15.0 |
| UN32FAPS | 11.0 | 9.8 | 11.1 | 10.2 |
| PACK 1 | | | | |
| PACK 2 | | | | |

*The shaded cells represent containers that leaked

### 5.6 Closure Back-Off and Package with Leaks

This section shows various pictures of containers that leaked after the various tests conducted in Phases I, II, III, IV, and V. In addition Figure 13 shows the test setup to monitor closure back-off between a bottle and cap. Figures 14 – 17 show some examples of UN approved packaging that leaked during various test phases. Figure 18 shows groups of caps that passed and failed during Phase I testing for packages obtained from Labelmaster Inc. In addition the bottle finish sections of Labelmaster Inc. packages were also checked for dimensional stability and out-of-round conditions after Phase I tests. This data is presented in Table 14.

There was no closure back-off recorded after the testing in all five phases. This is probably attributed to the fact that a secondary tape "seal" is applied on the closure and bottle after applying the torque to the closure. Also there was no significant "out-of-round" condition in any of the Labelmaster Inc. packages used in Phase I-V.

Table 15 shows the tests conducted on bottles and closure to measure the loss of torque on primary containers and closures that were used in the combination packaging during Phase I of the study over a period of seven days. The results showed that all bottles maintained residual torque at the end of the seven-day period.

**Table 14: "Out-of-Round" Dimensional Measurement for Bottles**

| SKU | "I" DIMENSION (mm) | | | | AVG. |
|---|---|---|---|---|---|
| | Reading 1 | Reading 2 | Reading 3 | Reading 4 | |
| HMS-08 | 34.15 | 34.15 | 34.15 | 34.15 | **34.15** |
| UN950PPT | 46.52 | 46.52 | 46.53 | 46.52 | **46.52** |
| UN950GPT | 42.43 | 42.43 | 42.43 | 42.43 | **42.43** |
| UN16FFPS | 18.47 | 18.47 | 18.47 | 18.47 | **18.47** |
| UN32FFPS | 24.63 | 24.63 | 24.63 | 24.63 | **24.63** |
| UNHWS16 | 59.22 | 59.22 | 59.22 | 59.22 | **59.22** |
| UN32NPVB | 52.03 | 52.03 | 52.03 | 52.03 | **52.03** |
| HMSP-32N | 28.05 | 28.05 | 28.05 | 28.05 | **28.05** |
| UN32PPS | 32.18 | 32.18 | 32.18 | 32.18 | **32.18** |
| UN4FFPS | 15.38 | 15.38 | 15.38 | 15.38 | **15.38** |
| UAC32FPS | 24.62 | 24.62 | 24.62 | 24.62 | **24.62** |
| UN32FAPS | 24.63 | 24.63 | 24.63 | 24.63 | **24.63** |
| | | | | | |
| **SKU** | **"T" DIMENSION (mm)** | | | | **AVG.** |
| | Reading 1 | Reading 2 | Reading 3 | Reading 4 | |
| HMS-08 | 43.46 | 43.46 | 43.46 | 43.46 | **43.46** |
| UN950PPT | 52.03 | 52.03 | 52.03 | 52.03 | **52.03** |
| UN950GPT | 51.02 | 51.02 | 51.02 | 51.02 | **51.02** |
| UN16FFPS | 27.03 | 27.03 | 27.03 | 27.03 | **27.03** |
| UN32FFPS | 31.16 | 31.16 | 31.16 | 31.16 | **31.16** |
| UNHWS16 | 68.44 | 68.44 | 68.44 | 68.44 | **68.44** |
| UN32NPVB | 62.28 | 62.28 | 62.28 | 62.28 | **62.28** |
| HMSP-32N | 37.29 | 37.29 | 37.29 | 37.29 | **37.29** |
| UN32PPS | 37.29 | 37.29 | 37.29 | 37.29 | **37.29** |
| UN4FFPS | 21.54 | 21.54 | 21.54 | 21.54 | **21.54** |
| UAC32FPS | 31.15 | 31.15 | 31.15 | 31.15 | **31.15** |
| UN32FAPS | 31.15 | 31.15 | 31.15 | 31.15 | **31.15** |

**Table 15: Loss in Torque Due to Creep**

| IMMEDIATE | | | 7 DAYS | | |
|---|---|---|---|---|---|
| AT | IRT | IRT/AT % | AT | RT | RT/AT % |
| 21.15 | 18.95 | 89.60 | 21.20 | 20.50 | 96.70 |
| 20.30 | 18.45 | 90.89 | 20.20 | 13.10 | 64.85 |
| 11.15 | 10.20 | 91.48 | 11.15 | 8.30 | 74.44 |
| 11.10 | 9.25 | 83.33 | 11.30 | 8.35 | 73.89 |
| 16.00 | 14.45 | 90.31 | 16.10 | 12.20 | 75.78 |
| 35.35 | 34.25 | 96.89 | 35.15 | 24.55 | 69.84 |
| 56.10 | 55.70 | 99.29 | 56.15 | 34.85 | 62.07 |
| 18.15 | 17.65 | 97.25 | 18.10 | 13.25 | 73.20 |
| 20.15 | 19.35 | 96.03 | 20.20 | 14.80 | 73.27 |
| 11.35 | 9.00 | 79.30 | 11.20 | 8.05 | 71.88 |
| 16.30 | 12.65 | 77.61 | 16.20 | 12.40 | 76.54 |
| 11.15 | 10.55 | 94.62 | 11.20 | 7.70 | 68.75 |

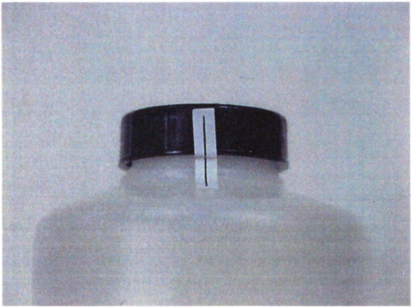


**Figure 13: Closure Back-Off Measurement**

**Figure 14: Phase I, HMS08, Labelmaster Inc.**


**Figure 15: Phase I, UNHWS16, Labelmaster Inc.**

**Figure 16: Phase I, UN32PPS, Labelmaster Inc.**

**Figure 17: Phase I, CT-SP-0002, CARGOpak Corp.**



**Figure 18: Labelmaster Inc. Caps that Passed/Failed Phase I Testing**

Figure 19, below, shows a comparison of leakage failures, as a percentage, for all five phases (for LABELMASTER Inc.). Clearly, phase I represented the highest (nearly 50%) number of leakers. Phase I results are similar with the observations of the FAA Office of Aviation Security. Phases II & III show that the current test procedures fail to show package failures as observed in real life. Phase IV shows that pressurized air shipments of approved packages show leaks based on the new test methods. Phase V shows that ground shipments at high altitudes (8000 ft) also show leaks on currently approved DOT packages.

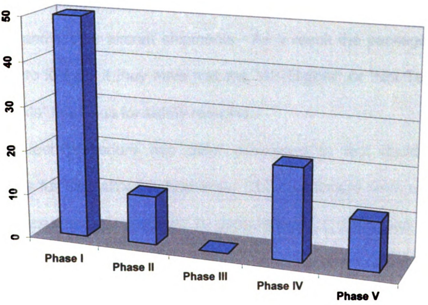



**Figure 19: Comparison of Leakage Failures (%) for the Five Phases of Testing (LABELMASTER Inc.)**

# 6.0 CONCLUSIONS

Based on the results of this study the following conclusions were made:

1. The existing test procedures used by US-DOT, UN, and ICAO do not prevent leaks from high altitude shipments of liquid hazardous materials.
2. The recommended test procedure to replicate high altitude shipments should include ***simultaneous*** vibration and low pressure environment.
3. Separate tests should be conducted on packages that are eligible for air and ground shipments based on the expected vibration levels and altitude pressure conditions described in Table 16.
4. There is a difference in the amount of leaks that occur in high altitude ground and feeder-aircraft shipments. As a result the package should be marked to identify if they have met the "Air-Eligible" or "Not Tested for Air Shipments" markings for safety reasons.
5. A new test procedure has been developed to test liquid hazardous packages for high-altitude shipments. This test should be in addition to all current tests being conducted in accordance with DOT and UN HazMat package requirements.

**Table 16: Pressure Conversion Table**

| Altitude, m | Altitude, ft | mm.Hg | In.Hg | kPa | psi |
|---|---|---|---|---|---|
| **0** | **0** | 760.00 | 29.92 | 101.3 | 14.70 |
| **305** | **1 000** | 732.90 | 28.85 | 97.7 | 14.02 |
| **1524** | **5 000** | 632.30 | 24.89 | 84.3 | 12.23 |
| **2438** | **8 000** | 564.85 | 22.24 | 75.3 | 10.92 |
| **3048** | **10 000** | 522.84 | 20.58 | 69.7 | 10.11 |
| **3658** | **12 000** | 483.83 | 19.05 | 64.5 | 9.35 |
| **4267** | **14 000** | 446.33 | 17.57 | 59.5 | 8.63 |
| **4877** | **16 000** | 411.82 | 16.21 | 54.9 | 7.97 |
| **5486** | **18 000** | 379.57 | 14.94 | 50.6 | 7.34 |
| **6096** | **20 000** | 349.56 | 13.76 | 46.6 | 6.76 |
| **7925** | **26 000** | 270.05 | 10.63 | 36.0 | 5.22 |
| **9144** | **30 000** | 225.60 | 8.88 | 30.1 | 4.36 |
| **12192** | **40 000** | 140.70 | 5.54 | 18.8 | 2.72 |
| **15240** | **50 000** | 87.30 | 3.44 | 11.6 | 1.69 |

## 6.1 Recommended Test Procedure for Liquid HazMat Shipments

*Test Procedure:*

- Pre-conditioned package samples at 73.4 ± 3.6°F for a minimum of 24 hours before testing
- The primary containers should be filled to the fill-level recommended and proper closure torque should be applied
- Apply secondary packaging, as if preparing for shipping, in accordance with the manufacturer's instructions
- Place the test specimen in a top-down or side-side orientation in the vacuum chamber and placed the vacuum chamber on an electro-hydraulic vibration table. Fasten the chamber to be table
- After sealing the vacuum chamber shut, turn the vacuum source on and adjust it to a rate of 305 meters (1000 feet) per 30-60 seconds as recommended in ASTM D6653-01.
- Use a vacuum of 59.5 kPa (pressure equivalent of 14,000 feet) for air-shipments and 69.7 kPa (pressure equivalent of 10,000) for ground shipments with a permissible error margin of ±2%
- While maintaining the required vacuum, operate the vibration table for 1 hour using random mode simulation with the Truck/Air Power Density Spectrum (Assurance level II, ASTM D 4169) for air shipments; or 3 hours using the Truck Composite Spectrum for ground shipments representing 1500 miles.

- At the end of the test, open the valve and release the vacuum at a rate of 305 meters (1000 feet) per 30-60 seconds
- Retrieve the sample packages and record any leaks

## 6.2 Air Eligibility Markings

Based on the findings of this study two new air-eligibilty markings were proposed to the ASTM Task-Group on pictorial markings at the 2002 ASTM Fall Meeting in Norfolk Virginia. These are shown in Figures 11 and 12. the task group approved these markings, which are now in Subcommittee and Main Committee ballot process.

**Figure 20: Air Eligible**

**Figure 21: Not Tested for Air Shipments**

# APPENDIX A

## A.1 The Hazardous Materials Table - Subpart B

*Column 2: Hazardous Materials Descriptions & Proper Shipping Names*

Lists hazardous materials descriptions and proper shipping names

- PSNs are in Roman type ONLY
- No alteration or modification allowed
- May use "N.O.S." as part of PSN when HazMat does not appear on HMT

***Authorized Shipping Names***

Four PSN Groups

- Group 1: Chemical Name

Examples: acetone, sulfuric acid, nitrogen

- Group 2: General Description

Examples: adhesives, paint-related materials, compounds, cleaning liquid

- Group 3: Generic names (Chemical Family)

Examples: alcohol, nitrates, insecticide gases

- Group 4: Hazard Class Names

Examples: flammable liquids, corrosive solids, compressed gases

- Words in *Italics* not part of PSN

- PSN can be singular/plural; CAPITALIZED or all lower case letters
- 

*Column 3: Hazard Class or Division*

- Contains hazard class designations that correspond to PSNs listed on HMT Mandatory
- "Forbidden" in this column means hazmat may not be transported by any means. It does not apply if hazmat is diluted, or incorporated in another product, or stabilized

*Hazard Classes*

- Hazard Classes Are Numbers
- Definitions in 49 CFR 173
- Hazard Must Be Right

*CLASS 1 – EXPLOSIVES*

§Explosives

- Divided into six (6) divisions
- Means any article designed to, or inherently capable of, extremely rapid release of gas and heat.

  Examples would include Trinitrophenol (Picric Acid) and Trinitrotoluene.


*CLASS 2 – GASES*

§Compressed Gases

- Divided into three (3) divisions
- Defined by temperature (68$^0$F) and pressure (14.7 psi) at which it becomes gas; and those additional characteristics (Corrosivity, flammability, etc.) which provide it's hazard characteristics

  Examples - oxygen, phosgene


*CLASS 3 - FLAMMABLE LIQUID*

§Flammable Liquids

- Liquid with flash point of not more than 141°F

  Examples - Paint thinner, Acetone, Methanol.


*CLASS 4 - FLAMMABLE SOLIDS*

§Flammable Solid

- Divided into three (3) divisions
- Division 4.1: Flammable Solids
- Division 4.2: Spontaneously Combustible
- Division 4.3: Dangerous When Wet

  Examples - Sodium Metal.


*CLASS 5 - OXIDIZING SUBSTANCES*

§Oxidizers

- Divided into two (2) divisions

- Division 5.1: Oxidizers
- Division 5.2: Organic peroxides

  Examples - Benzoyl Peroxide, Sodium Nitrate.


*CLASS 6 – POISON*

§Poisonous Materials

- Divided into two (2) divisions
- Division 6.1: Poison (Other than Gas)
- Division 6.2: Infectious substances

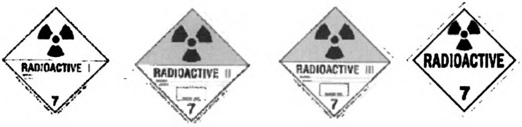
  Examples – Cyanides


*CLASS 7 - RADIOACTIVE MATERIALS*

§Radioactive Materials

- Considered acutely hazardous substances
- Restricted by packaging, quantity, labeling & marking, routes of transport, means of transport

- Also controlled by NRC




*CLASS 8 - CORROSIVE MATERIALS*

§Corrosives

- A liquid/solid that causes full thickness destruction of human skin at point of contact
- A liquid that has a severe corrosion rate on steel or aluminum

  Examples - Acids, Bases


*CLASS 9 - MISCELLANEOUS HM*

§Miscellaneous Hazardous Materials

- Material presents hazard during transport but doesn't meet definition of any other hazard class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## A.2 How to Read a UN Number or Marking

The marking that is applied to a UN certified package indicates the type of package and the levels to which the packaging has been approved. The following describes the sequence of numbers and letters that appear in a UN marking and what they designate.

### *Contents of UN Markings*

The markings associated with performance criteria indicate the type of package and the levels to which the package has been approved. Each set of information is separated by a slash mark (/). The following explains each set of numbers and letters in the sequence.

***UN Indication*** - The package must be marked with a UN Symbol, or just the letters UN are required on embossed metal containers.

***Packaging Identification Code*** - This code identifies the type of packaging, the material of construction, and a category within the type when applicable.

## Packaging Identification Table

| Type of Package | Material | Category |
|---|---|---|
| **1** - Drums | **A** - Steel | **<u>A, B, or H Drums-Jerricans</u>** |
| | | **1** - Closed Head |
| **2** - Barrels | **B** - Aluminum | **2** - Open Head |
| **3** - Jerricans | **C** - Natural Wood | **<u>A or B Boxes</u>** |
| **4** - Boxes | **D** - Plywood | **1** - Ordinary **A** or **B** |
| | | **2** - **A** or **B** w/inner lining or coating |
| **5** - Bags | **F** - Reconstituted Wood | |
| | | **<u>C Boxes</u>** |
| **6** - Composite Packagings | **G** - Fiberboard | **1** - Ordinary |
| | | **2** - w/sift proof walls |
| | **H** - Plastic | |
| | | **<u>H Boxes</u>** |
| | **L** - Textile | **1** - Expanded Plastic |
| | **M** - Paper, Multiwall | **2** - Solid Plastic |
| | **N** - Metal other than Steel | **<u>L Bags</u>** |
| | xxx or Aluminum | **2** - Sift proof |
| | | **3** - Water Resistant |
| | **P** - Glass, Porcelain or | |
| | xxx Stoneware | **<u>M Bags</u>** |
| | | **2** - Multi-wall, Water |
| | | xxx Resistant |

Example: The Packaging Identification code 1H1 would indicate a drum, made of plastic, with a closed-head configuration.

***Performance Standard Code*** - This code identifies the packing group(s) that the package has been tested and approved for.

X for Packing Groups I, II, and III

Y for Packing Groups II, and, III

Z for Packing Group III only

***Relative Density (Specific Gravity) or Gross Mass*** - A designation of Specific Gravity or Gross Mass for which the packaging has been successfully tested should follow the Performance Standard Code.

a. Stand-alone packagings intended to contain liquids must be marked with the specific gravity rounded down to the first decimal.
b. Packagings intended for solids or that have inner packagings must be marked with the maximum gross mass (weight) in kilograms.

***Designation of "S" for Solids or the Hydrostatic Pressure Test Rating in Kilopascals*** - An "S" in upper case should follow the gross mass to designate that the package is only intended for solids or inner packagings. Single or Composite packagings intended for liquids should reflect the Hydrostatic test pressure in kPa (kilopascals), rounded down to the nearest 10 kPa.

***Year of Manufacture*** - The last two digits of data indicate the year the packaging was manufactured.

## Examples of UN Markings

***Square Plastic Tighthead Pail*** 3H1/Y1.8/200/94USA/+AA0089

| | | |
|---|---|---|
| 3 = | Jerrican (square container) | **Type of Package** |
| H = | Plastic | **Material** |
| 1 = | Closed-Head | **Category** |
| Y = | Packing Group (II) | **Performance Standard Code** |
| 1.8 = | Maximum Specific Gravity of Product | **Relative Density** |
| 200 = | Kilopascals (kPa), also referred to as PSI | **Hydrostatic Pressure Rating** |
| 94 = | Year container was produced | **Year of Manufacture** |
| USA = | Marked under authority of USA | |
| +AA0089 = | Testing lab identification and test number of container | |

***Round Openhead Steel Pail*** UN1A2/Y23/S/93USA/+AA1234

| | | |
|---|---|---|
| 1 = | Drum (round) | **Type of Package** |
| A = | Steel | **Material** |
| 2 = | Open-Head | **Category** |
| Y = | Packing Group (II) | **Performance Standard Code** |
| 23 = | Weight in kilograms | **Gross Mass** |
| S = | Tested for Solids | **Solids** |
| 93 = | Year container was produced | **Year of Manufacture** |
| USA = | Marked under authority of USA | |
| +AA1234 = | Testing lab identification and test number of container | |

## *Combination Packaging with 2 Metal Paint Cans as Inner Packagings*

4G/Y10.4/S/94USA/+AX 1259

| | | |
|---|---|---|
| 4 = | Box | **Type of Package** |
| G = | Fiberboard | **Material** |
| Y = | Packing Group (II) | **Performance Standard Code** |
| 10.4 = | Weight in Kilograms | **Gross Mass** |
| S = | Designates Inner Packagings | **Solids or Inner Packagings** |
| 94 = | Year package was produced | **Year of Manufacture** |
| USA = | Marked under authority of USA | |
| +AX 1259 = | Testing lab identification and test number of container | |

## A.3 HAZARDOUS MATERIALS PACKAGING GLOSSARY

**CFR-49 (Code of Federal Regulations - Transportation)** A codified set of regulations formulated by the U.S. Dpartment of Transportation (DOT) governing the packaging and shipping of hazardous materials. Latest revision is October 1, 1996.

**COMBINATION PACKAGING**

One or more inner packagings used in combination with a non-bulk outer packaging. This does not include a Composite Packaging.

**COMPOSITE PACAGING**

A packaging consisting of an outer packaging and an inner receptacle. It is constructed so that the inner receptacle and outer packaging form an integral packaging. Once assembled it remains a single unit and is filled, stored, transported, and emptied as such.

**D.O.T.**

Department of Transportation.

**HM-181**

A set of the proposed new packaging and shipping regulations which since have been incorporated into CFR-49. This document is no longer applicable.

**HAZARD CLASSIFICATION**

Materials are grouped as to the specific hazard they present. The groups are Explosives, Gases, Flammable Liquids, Flammable Solids, Oxidizers, Poisonous Materials, Corrosive Materials and Miscellaneous.

**HAZARDOUS MATERIAL**

A substance having properties capable of having adverse affects on the health or safety of individuals.

**HAZARDOUS MATERIALS TABLE**

An alphabetical listing of the hazardous materials found in CFR-49, section 172.101. It lists the product by proper shipping name, and its UN number. It lists the hazard classification, packing group, and the sections in CFR 49 that apply to the packaging and shipping of a specific product.

**INNER PACKAGING**

A packaging for which an outer packaging is required. This does not include the inner receptacle of a composite packaging.

**JERRICANS**

Metal or plastic containers of rectangular or polygonal cross-section.

**LIMITED QUANTITY**

The quantity of hazardous material that may be shipped in packaging that is not UN certified. The quantity will vary depending on the specific product shipped, the mode of transportation, and the country the shipping occurs.

**MASS**

The maximum combined mass (weight) of inner packagings, or single packagings intended for solids, and the contents thereof.

**MSDS**

Material Safety Data Sheet. It is provided by manufacturers of hazardous materials, and describes the properties and nature of the material.

**OUTER PACKAGING**

The outermost packaging or enclosure of a combination or composite packaging along with any other cushioning or absorbent material and other components necessary to protect and contain inner packagings or receptacles.

**OVERPACK**

An enclosure used to provide protection or convenience in handling of a package or to consolidate two or more packages. The package being overpacked must be eligible to be transported by itself, and properly prepared for shipment with the proper markings and labeling. The marking and labeling on each of the packages being overpacked must be reproduced on the outside of the overpack unless visible from outside of the overpack.

**PACKAGE**

The end result of the packaging process, which includes all of the hazardous contents, and all of the packagings properly closed and prepared for proper marking and labeling.

**PACKAGING**

Containers, receptacles and all components necessary for the container or receptacle to perform its containment function and meet the requirements of CFR 49, parts 171-180. In general, these receptacles and components and other requirements are contained within CFR 49, part 173.

**PACKING GROUP**

The degree of hazard. Within each hazard classification there are three packing groups (I, II, and III). Packing Group I represents the greatest hazard, Group II a moderate hazard, and Group III the least hazard. In the marking of packagings, Group I corresponds to "X", Group II corresponds to "Y", and Group III corresponds to "Z".

**PERFORMANCE ORIENTED PACKAGING**

A set of criteria establishing the acceptability of a packaging to be used for hazardous materials based on its performance in established test procedures.

**SINGLE PACKAGING**

A single receptacle into which material is loaded other than a combination or bulk packaging. A drum is an example of a single packaging.

**TORQUE TEST**

A test designed to ascertain the stiffness of a material under given environmental conditions.

**UN MARKING**

The marking applied to a certified packaging indicating the Packing Group, and the severity of the testing performed.

**UN PACKAGING**

A packaging approved and certified for hazardous materials that has passed all required performance tests.

## UN RECOMMENDATIONS

A set of recommendations proposed by the U.N. Panel of Experts regarding the packaging and shipping of hazardous materials. These are only recommendations, but have been incorporated into the regulations of most countries and carrier organizations. They form the basis of HM-181 and the changes to CFR-49.

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