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Nondestructive Examination of Plutonium-Bearing Material Containers

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Abstract

The first nondestructive examination (NDE) of 3013-type containers as part of the Department of Energy's (DOE's) Integrated Surveillance Program (ISP)[1] was performed in February, 2005. Since that date 280 NDE surveillances on 255 containers have been conducted. These containers were packaged with plutonium-bearing materials at multiple DOE sites. The NDE surveillances were conducted at Hanford, Lawrence Livermore National Laboratory (LLNL), and Savannah River Site (SRS). These NDEs consisted of visual inspection, mass verification, radiological surveys, prompt gamma analysis, and radiography.

The primary purpose of performing NDE surveillances is to determine if there has been a significant pressure buildup inside the inner 3013 container. This is done by measuring the lid deflection of the inner 3013 container using radiography images. These lid deflection measurements are converted to pressure measurements to determine if a container has a pressure of a 100 psig or greater. Making this determination is required by Surveillance and Monitoring Plan (S&MP)[2]. All 3013 containers are designed to withstand at least 699 psig as specified by DOE-STD-3013[3]. To date, all containers evaluated have pressures under 50 psig. In addition, the radiography is useful in evaluating the contents of the 3013 container as well as determining the condition of the walls of the inner 3013 container and the convenience containers. The radiography has shown no signs of degradation of any container, but has revealed two packaging anomalies.

Quantitative pressure measurements based on lid deflections, which give more information than the "less than or greater than 100 psig" (pass/fail) data are also available for many containers. Statistical analyses of the pass/fail data combined with analysis of the quantitative data show that it is extremely unlikely that any container in the population of 3013 containers considered in this study (e.g., containers packaged according to the DOE-STD-3013 by 2006) would exceed a pressure of 100 psig. At this time, Los Alamos National Laboratory (LANL) and LLNL continue to package containers. Future NDE surveillances will address containers packaged after 2006 for both sites as well as containers requested by the Materials Identification Surveillance (MIS) working group based on knowledge gained from shelf-life study and surveillance results.

Introduction

The first ISP NDE was performed February 27, 2005 in the F-Area Material Storage Facility at the SRS. Since this time, 255 3013-type containers have undergone NDE at Hanford, LLNL, and SRS. These containers were packaged at multiple DOE sites, including Hanford, LLNL, Rocky Flats Environmental Technology Site (RFETS), and SRS.

The S&MP sampling specification includes binning the population of containers into three bins based on a container's contents and assumed potential for experiencing selected degradation mechanisms.[4,5] Potential degradation mechanisms include corrosion and pressurization. The three bins have been designated: Pressure and Corrosion (pressurization and corrosion mechanisms possible), Pressure Only (pressurization only, corrosion unlikely) and Innocuous (pressurization and corrosion unlikely). A random sample of 3013 containers is selected from each bin with containers proportionally selected from each packaging site. The random sample is augmented with containers selected based on the engineering judgment of the MIS working group. In addition, the program allows the packaging sites to perform NDE on 3013s to address facility specific concerns. Table 1 shows the allocation of the 255 NDE containers to the three bins (Pressure and Corrosion, Pressure Only, and Innocuous) and identifies the sample selection criterion (random, judgmental, and additional).

Nondestructive Examinations (NDEs)

The surveillances performed to verify the integrity of the 3013 containers includes the following: contamination survey, visual inspection, mass verification, and full container radiography of the 3013 container and its contents.

A radiological survey is required for radiological protection purposes prior to handling the 3013 container. The survey also provides verification that at least one of the containers in the 3013 container set is intact. The DOE-STD-3013 requires that the outside of the inner 3013 container is free of radioactive contamination at the time of packaging, thus, any indication of radiological contamination on the external surface of the outer 3013 container could be an indication of penetration of both the inner and outer 3013 container walls. Of the 3013 containers that have undergone NDE to date no contamination on the external surface of the outer 3013 has been detected.

A visual inspection of the external 3013 container surface is performed to identify both breaches in the outer container and conditions which could lead to a breach in the outer container. None of the 3013 containers that have undergone NDE surveillance have had a breach in the outer container, or any evidence of pitting corrosion, large dents, scratches, or other conditions adverse to quality on the outer 3013 container surface.

The 3013 container mass determinations monitor for changes in the mass of the 3013 container/package system. The purpose of the mass monitoring include verifying the as-packaged system mass and determining if any changes in the mass could indicate a failure in the 3013 container integrity. For example, over time plutonium metal will be converted to plutonium oxide if exposed to air and the calcined plutonium oxide may adsorb moisture from the air. These effects would result in a weight gain of the 3013 container. No 3013 container evaluated by the Surveillance Program has failed the mass verification. At the Savannah River Site, this means the mass of all 3013 containers at the time of surveillance was within one gram of the buoyancy corrected baseline mass measurement. A buoyancy correction is needed to correct for the difference between the air density between packaging site (RFETS) and SRS due to the difference in elevation. For example, the buoyancy corrected baseline mass is typically approximately 0.4 grams less than the RFETS measured baseline mass. In fact, the majority of the 3013 containers measured have been within 0.2 grams of the buoyancy corrected baseline mass.

Full container radiography is used as part of NDE at the SRS and LLNL to examine the contents of the 3013 container. At LLNL, the surveillance is performed using film. At the SRS, digital radiography is used to obtain full container (composite) images at 0º and 90º. Full container radiography is useful as part of an NDE program, as it allows some conclusions about the condition of the inner 3013 and any convenience cans present. This is done by examining the air gap between the outer and inner 3013 container and between the convenience container and inner 3013 container for the presence of nuclear material. If no material is observed in these air gaps, the container walls are most likely intact. The full can radiography performed to date has not revealed any 3013 containers with nuclear material in the gaps between the various nested containers. It should be noted that the majority of the packaging sites did not perform full container radiographs, but only radiographed the lid of the inner container to document the initial lid location to use for future pressure measurements. LLNL is the only packaging facility that performed full container radiography.

Full container radiography has identified two 3013 containers with content anomalies. In both of these cases, the subject 3013 container subsequently underwent Destructive Examination (DE). In the first case, a metal scoop used for sampling the oxide at the packaging site (Figure 1) was found in the oxide material. The full container radiography image (Figure 2) clearly shows the presence of a piece of high density material (metal) protruding above the surface of the bulk oxide material.

During a later NDE surveillance of another 3013 container, the field surveillance engineer observed an unusual object and made notifications to the surveillance program. Further review was performed, including taking images at additional angles. The 3013 container was also shaken and rotated by the operator followed by additional images to observe any changes. The full container radiograph clearly shows darker material (Pu oxide) above the surface of the bulk oxide suspended in some low density material (indicated by the light wispy areas of the image) (Figure 3). DE of this 3013 revealed that a plastic glove (Figure 4) had been inadvertently left in the 3013 during packaging operations. It should be noted that in both of these cases, the tramp material extended above the surface of the bulk oxide. If it had been completely submerged within the bulk oxide, full container radiography may not have been able to distinguish the material. Based on this, full container radiography can indicate the presence of tramp material if it extends above the oxide surface, but the failure to see an anomaly in an image of a container does not conclusively prove that tramp material is not present. Review of the original packaging data for these containers revealed that both containers had small weight discrepancies. As a result, the MIS Working Group identified 18 Hanford containers that have potential weight discrepancies and requested that SRS perform DE on two containers and NDE on the remaining 16 containers. To date, SRS has completed DE on both of the identified containers and NDE on 10 of the identified containers. No foreign material was found in the DEs, but two of the NDEs have indicated that a scoop is present.

Pressurization Measurements

Historically, container pressurization in plutonium bearing materials in convenience cans was measured through the use of mechanical lid deflection measurements. However, in the 3013 container configuration, the deflecting component is the inner 3013 container lid, which is inaccessible without the destruction of the outer 3013. Therefore, radiography was selected to determine the amount of lid deflection in a 3013 inner container. DOE-STD-3013 requires a baseline measurement be performed within 30 days after welding of the container. The Standard also requires that the container and radiography system be capable of detecting a pressure increase of 100 psig or greater. Therefore, all packaging sites are required to be able to perform radiography on 3013 containers. Each packaging site has some degree of flexibility on the configuration of the inner 3013, as well as on how radiography is performed. The Savannah River National Laboratory developed a method to correlate the amount of lid deflection with the amount of pressure in the 3013 container[6,7,8] and the associated uncertainties. The ADRIS (Automated Digital Radiography Inspection System) was developed at SRS. It is based around an industrial 440 KV X ray unit. It uses digital radiography to measure the amount of lid deflection, compare it to the measured baseline deflection and correlate this difference to the change in pressure in the 3013 container.[6,7,8]

RFETS and LLNL used inner 3013 containers with a relatively thin lid. The thin lid deflects easily, allowing changes in the lid deflection to be readily observed. RFETS used a digital radiography system to establish the baseline radiographs, while LLNL used film radiographs. The scanned LLNL radiography images do not support quantitative pressure assessments using ADRIS, however, studies show that LLNL lids exhibit “snap through” between 20 psig and 30 psig.[6] Thus the LLNL data are “pass/fail” data with a trigger point well below 100 psig.

The Hanford Plutonium Finishing Plant and SRS FB-Line used a thicker lid on the inner container. Digital radiography for lid deflection measurements at Hanford was performed using the ADRIS . Baseline measurements were taken within 30 days of container closure and at 30, 60, and 90 day intervals for many containers. Over 2000 radiographs were taken. These initial efforts uncovered problems with accurately determining the lid deflection for containers with severely tilted inner lids. The “shadow” from the edge of the tilted lid would obscure the true center lid deflection point. Called the “dead zone”, a joint SRS/Hanford resolution of this issue resulted in several findings:[9]

- The standard 4 view inspection program (view at every 90 degrees) was adequate for most cans.
- An optimal viewing angle exists, unique to each container, where the field of view for lid deflection measurement is minimally affected by lid edge shadow. Special inspection programs for these cans were developed.
- For cans with baseline measurement in this “dead zone”, the digital micrometer measurement could serve as the baseline measurement and equivalency methods for comparing micrometer measurement to digital radiograph were developed.
- Equivalency in measurement for all three digital radiograph systems in use (one at Hanford, two at SRS) was demonstrated.

FB-Line addressed the dead zone issue by revising the design of the inner 3013 lid to include a “button” on the center portion of the inner 3013 lid bottom. The button was shaped to preclude the creation of a dead zone. (Figure 5)

NDE at the SRS addresses the dead zone issue for Hanford 3013s by using the optimal viewing angle. This angle is calculated based upon the radiograph images at the four standard viewing angles. A radiograph is taken at both the optimal viewing angle and 180 degrees away from the optimal viewing angle. This image is compared to the closest Hanford standard view baseline and correlated to a pressure. However, there are clearly larger pressure measurement uncertainties associated with Hanford containers with dead zone problems than with other containers.[10]

One 3013 at Hanford was discovered to have been packaged in excess of the standard specification of 0.5wt% water during a thorough QA review of all containers. Radiography was performed on this container and the results showed a slight positive detection of lid deflection, attributed to be from 35-50 psig. The NDE findings on this item were instrumental in demonstrating the ability of NDE to detect pressurization and in assuring that the container could continue in safe storage until the development of the technical basis to allow shipment offsite was completed. This item is currently scheduled for destructive analysis and further evaluation by LANL in 2010.

Statistical Analysis of Pressurization Data

The data used in this analysis include 252 containers with lid deflection pressurization measurements (LDF psig) measurements (note that there are 252 rather than 255 because the three LLNL containers have only pass/fail data, thus they are not included). In the case of multiple measurements on a single container, the most recent measurement was selected from the larger database, as this is considered the best measurement to accurately reflect pressurization. In addition to the LDF psig measurements, there are 110 containers evaluated at SRS that have calibration-based standard deviations for the measurements (sigmas). The containers undergoing DEs also have gas measurements taken at the time of the DE using the Gas Evaluation Software Tool (GEST).[11] There are 43 containers with GEST data.

The ISP sampling design (described in references 4 and 5) requires 130 randomly selected containers from the Pressure Only bin. These 130 are spread proportionately across the sites that have generated containers by 2006 – Hanford, LLNL, RFETS and SRS. The specification of 130 containers is based on the criterion that if pass/fail data (less than 100 psig/greater than 100 psig) are available, then, if no containers in the 130 observations are greater than 100 psig, the conclusion is that there is 99.9% confidence that not more than 5% of the entire Pressure Only bin population could have a pressurization greater than 100 psig. The sampling criterion is met and the conclusion follows for the Pressure Only bin.

In addition to the pass/fail data, quantitative pressure measurements (LDF psig) exist for all containers except those from LLNL. Figure 6 compares these data across bins (box plots[i] are used for the comparison) and Figure 7 compares these data across the sample selection criterion (random, judgmental, and additional). These figures show that LDF psig measurements do not differ significantly between the various groupings, therefore, these data can be combined across bins and sample selection criterion to obtain more reliable results.

Figure 8 compares the LDF psig data across packaging sites. This figure shows that the Hanford data have more outliers than the other sites. Note that the highest LDF psig measurements are identified as potential outliers in the Hanford data. These outliers are likely due to measurement errors resulting from dead zone measurement problems.[10] However, even these high measurements are under 50 psig.

A comparison of the distribution the LDF psig measurements to a normal distribution using a Q-Q plot[ii] shows that the outliers in the Hanford data result in tails (the largest and smallest data values) that deviate significantly from those of a normal distribution (Figure 9). When the Hanford data (except for the Innocuous bin measurements) are removed, the comparison to a normal distribution is much better

(Figure 10). In this case, the lower tail falls below the normal line, however, it is only the upper bound on the population that is of interest. Using the normal distribution for the upper tail is a reasonable assumption for these data. Applying normal distribution theory to this reduced data set, one finds that there is 99.5% confidence that 99.99% of the population is less than 42 psig (the 99.5%/99.99% upper tolerance limit [UTL]).[12] This result indicates that it is unlikely that a LDF psig measurement from RFETS or SRS would exceed 42 psig. Consequentially, it is exceedingly unlikely that a measurement would exceed 100 psig. Although the Hanford data outliers do not permit using normal theory to get tolerance limits, it is useful to note that it would take almost seven standard deviations from the mean of the Hanford data to get to 100 psig (Table 2 contains the mean and standard deviation).

Many of the container lid deflection pressure measurements have an associated sigma (standard deviation) based on the calibration results.[6,7,8] These sigmas vary from 3 psig to almost 9 psig. The sigmas are based on experimental conditions in the laboratory and it is highly probable that they underestimate the actual measurement uncertainty occurring in the field (particularly for Hanford containers with dead zone issues). Table 2 shows the means, standard deviations, number of containers and 95% confidence intervals for the means calculated from the field data for each packaging site. RFETS and SRS standard deviations are within the range of calibration measurement errors, indicating that the main source of variability is from measurement error with negligible variability between containers. Hanford has a larger standard deviation than seen in the calibration measurement sigmas, but it is the outliers causing this variability and these outliers are likely a result of dead zone measurement problems.

Containers undergoing destructive examinations (DE) have actual gas measurements (GEST) collected at the time of the DE.[11] There are 43 containers with GEST measurements, 20 from Hanford and 23 from RFETS. These GEST measurements provide the best possible assessment of pressurization. For the containers with GEST measurements the LDF psig can be compared to the GEST (adjusted for atmospheric pressure at the packaging site (e.g., GEST-11.8 for RFETS and GEST-14.4 for Hanford). These differences provide an estimate of the bias of the field LDF psig measurements. The RFETS bias is positive (5.2 psig) with 95% confidence bounds, (3.8, 6.7) psig. This positive bias shows that the LDF psig measurements for the RFETS data are conservative, e.g., overestimate the actual pressure by a small amount. The Hanford bias is -1.6 psig with 95% confidence bounds (-9.3, 6.1) psig. Although the bias confidence bounds bracket zero, there is so much scatter in the data it is impossible to draw conclusions other than that the Hanford biases are quite variable and can be both negative and positive.

The sampling specification for the Innocuous bin was to evaluate the assumption of no pressurization and essentially no variability between containers.[4,5] However, because the main source of variability is measurement variability, it is not possible to definitively evaluate this assumption. Nevertheless, as shown in Figure 6, the Innocuous bin items do not look significantly different from the other bins. In this analysis the Innocuous bin containers are included in the calculation of the 99.5%/99.99% UTL of 42 psig.

Conclusions

Nondestructive examination surveillances of two hundred and fifty-five 3013 containers have not identified any conditions challenging the 3013 container integrity. Pressure measurements based on lid deflection measurements from digital radiography images (LDF psig) have not indicated any appreciable pressurization in any of these 255 containers. Comparison of LDF psig measurements to actual gas measurements made during destructive examinations of RFETS containers indicates that the LDF psig measurements are conservative (e.g., slightly overestimate pressure changes) for the RFETS containers.

The 3013 Standard requires containers be designed to withstand 699 psig of internal pressure, but the S&MP set a normal acceptance criterion of 100 psig during surveillance. Using the "greater than 100 psig" criterion as a trigger for pass/fail decisions, the conclusion from the 130 randomly selected Pressure Only bin samples is that there is 99.9% confidence that a container having a pressure above 100 psig would not occur in more than 5% of the population of containers from Hanford, LLNL, RFETS, and SRS packaged by 2006. In addition to the pass/fail data, there are 252 pressure LDF psig measurements. All of these measurements are under 50 psig.

Comparison of field LDF psig measurement variability based on the NDE surveillance data to LDF psig variability estimated from laboratory controlled calibration experiments indicates that the main source of variability in the field LDF psig measurements for RFETS and SRS is from the measurement process. The Hanford data have many potential outliers (likely from dead zone measurement problems) that result in variability greater than the laboratory calibration results. However, it is unlikely that any containers exceed 100 psig.

The RFETS and SRS data can be used to develop an upper tolerance limit for the general population of 3013 containers packaged by 2006 and following the specifications of DOE STD 3013[iii]. (The large measurement errors seen in the Hanford data require removing these data from the analysis data set used to develop the upper tolerance limit.) The upper 99.5%/99.99% tolerance limit based on the remaining data is 42 psig (this means that there is 99.5% confidence that 99.99% of the population is below 42 psig). This finding makes it extremely unlikely that RFETS or SRS containers would exceed 100 psig. Even though the Hanford data have increased variability due to dead zone measurement issues, it requires approximately seven standard deviations from the mean to reach 100 psig for these data.

Based on these results it appears that there is little or no pressurization occurring in the 3013 containers and that variability in the data is a result of inherent measurement variability rather than container-to-container variability.

Future NDE surveillance will be guided by these NDE findings. For example the discovery of anomalies in Hanford packaging has focused NDE surveillance on Hanford containers with weight discrepancies. Future NDE surveillances will also evaluate containers generated after 2006 from LANL and LLNL.

NOTES

i. A box plot provides an excellent visual summary of many important aspects of a distribution and allows quick comparison between data sets. The box stretches from the 25th percentile to the 75th percentile (middle half of the data). The median is shown as a line across the box. The "inter-quartile range" is the difference between the top and bottom of the box. The whiskers come out from the top and bottom of the box (lines with tops) to the first data points that are not beyond 1.5*the inter-quartile range. These are either the max and min of the data, or, if points lie beyond these, the max and min of the data will be the top and bottom filled circles. The points outside the whiskers are considered potential outliers.

ii. Quantile-Quantile (Q-Q) plots (in this analysis) compare the quantiles of the data with the quantiles of the normal distribution. If the data are normal they will lie approximately along the line (the normal line).

iii. Tolerance limits specify a region that covers a certain portion of the population (e.g., 99.99%) with a certain level of confidence (e.g.,99.5%). Tolerance limits can also be viewed as confidence limits on population percentiles.

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Figure 1: Spatula Pitcure

Figure 2: Full can radiography image showing spatula

Figure 3: Full can radiography image showing glove

Figure 4: Picture of Glove Tramp Material

Figure 5: FBL 3013 Can Configuration with Button on Inner 3013 Lid

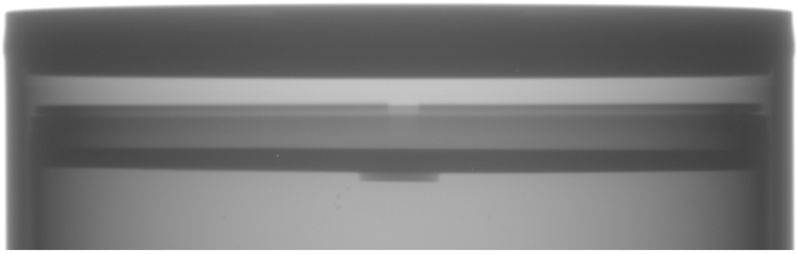


Table 1. NDE on Containers broken out by site, bin and type

Site	Innocuous Bin	Pressure Only Bin	Pressure and Corrosion Bin	Total
Hanford total	4 (R) 4	63 (R) 5 (J) 9 (A) 77	22 (R) 10 (J) 29 (A) 61	 142
LLNL	---------------	1(R)	2 (R)	3
RFETS total	4 (R) 4	58 (R) 3 (A) 61	21 (R) 13 (J) 1 (A) 35	 100
SRS	2 (R)	8 (R)	----------	10
Sub Totals	10 (R)	130 (R) 5 (J) 12 (A)	45 (R) 23 (J) 30 (A)	
Total	10	147	98	255

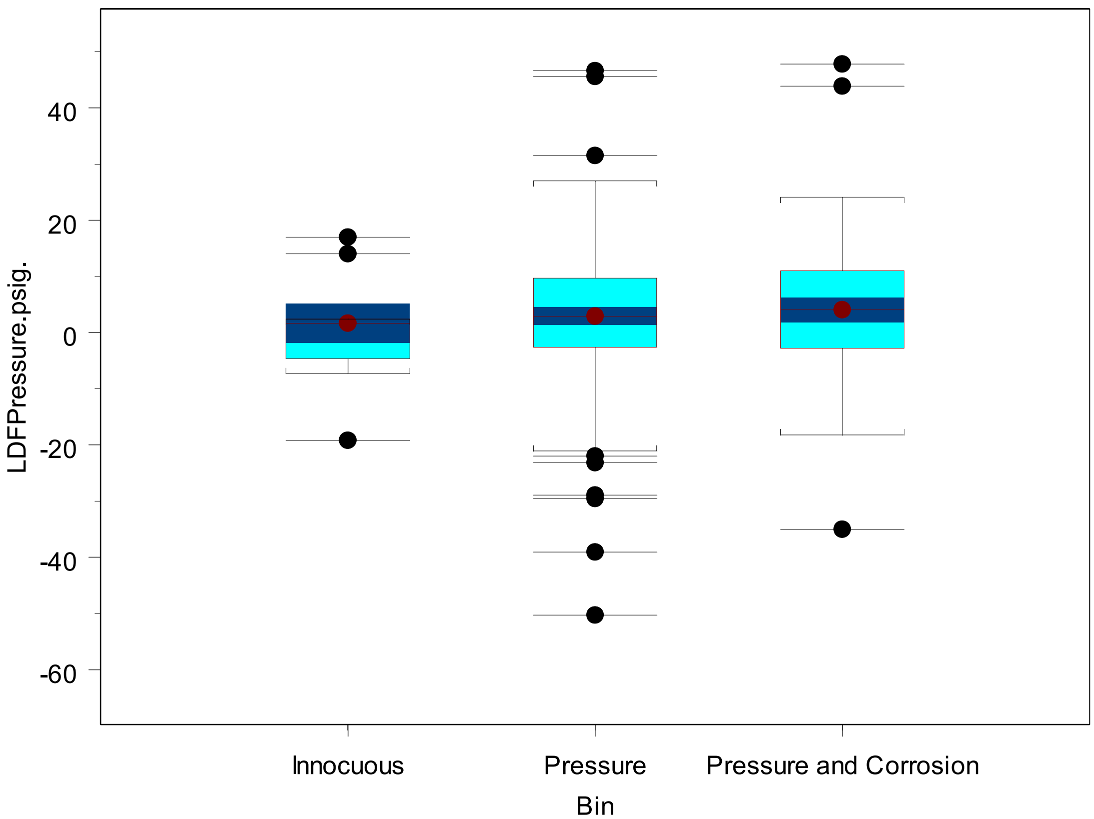
R = Random, J = Judgmental, and A = Additional

Table 2. Means, standard deviations (SD), number of containers (N), and 95% confidence intervals (CI)

Site	Mean	SD	N	CI
Hanford*	-0.27	14.4	138	(-2.7, 2.2)
RFETS*	9.2	6.0	96	(8.0, 10.4)
SRS*	-5.1	5.7	8	(-9.9 ,-0.3)
RFETS & SRS (with Innocuous)	7.4	7.7	114	(6.0, 8.9)

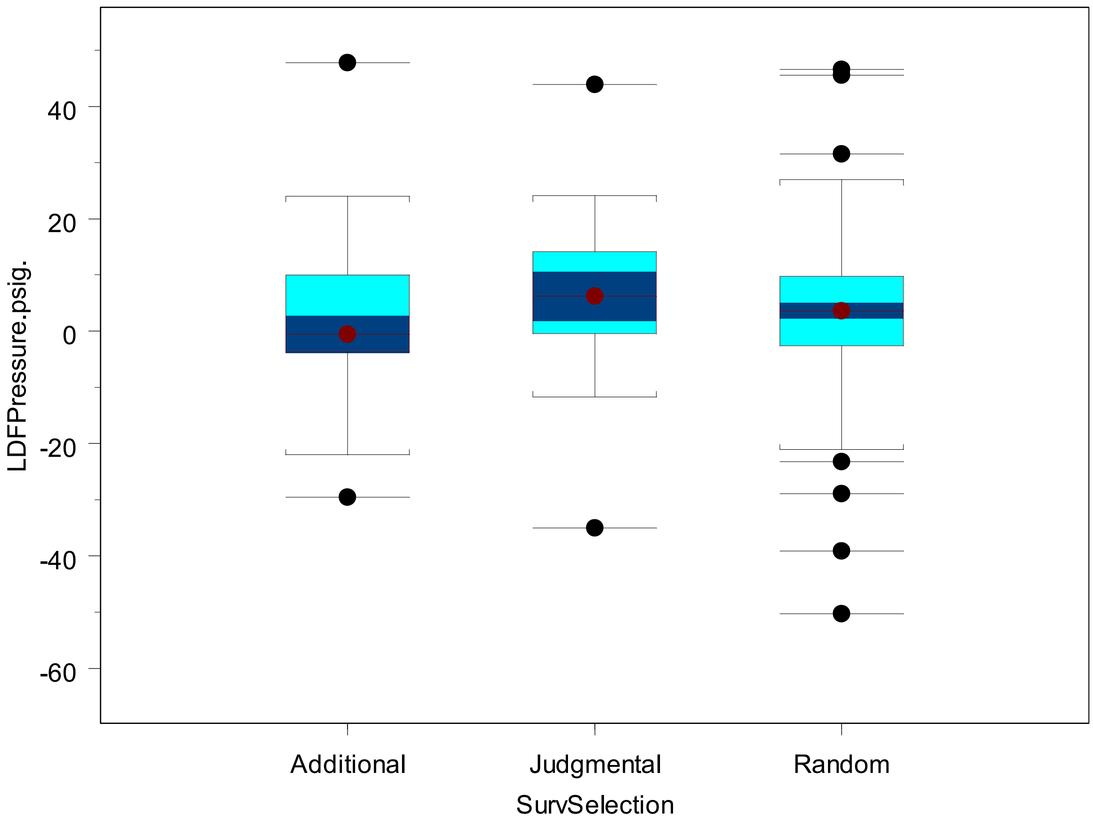
* Containers from the Innocuous bin are not included.

Figure 6. Box plots of the LDF psig measurements for each bin.



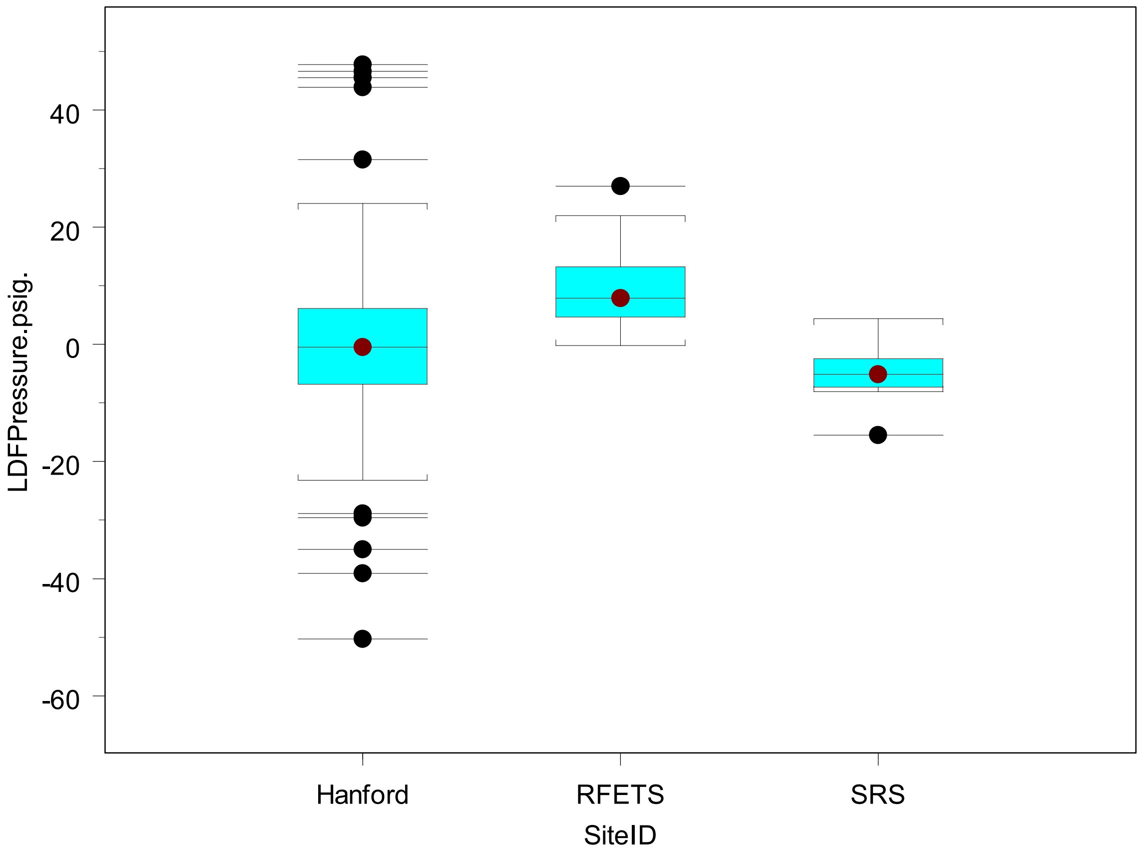
The dark color contains the 95% confidence limits on the mean. The lines with filled circles identify measurements that differ significantly from the rest of the population and could be outliers. There are no significant differences between the means of these populations. The spread of measurements varies between bins, but this is expected since the Innocuous bin has only 10 observations.

Figure 7. Box plots of the LDF psig measurements for the various sampling selection criteria.



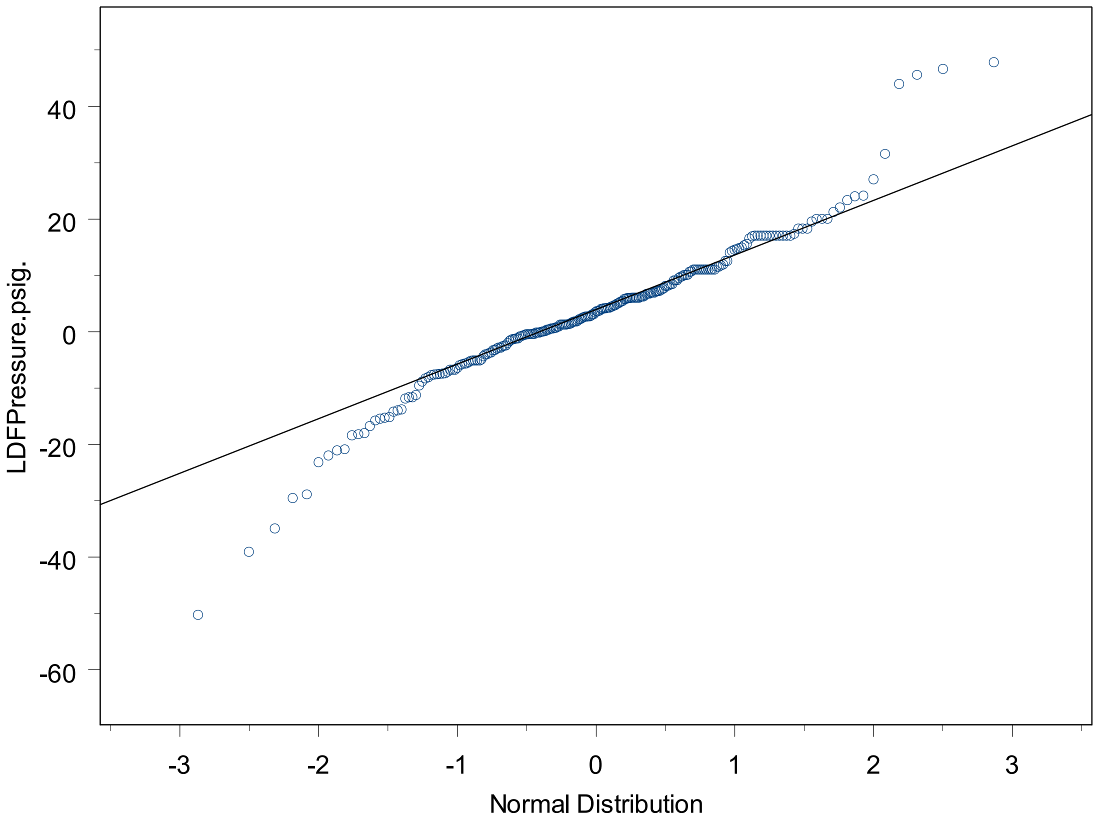
There are no significant differences between the means of these populations and the spread of the data look equivalent given the varying sizes of the populations.

Figure 8. Box plots of the LDF psig measurements for the three packaging sites, Hanford, RFETS and SRS



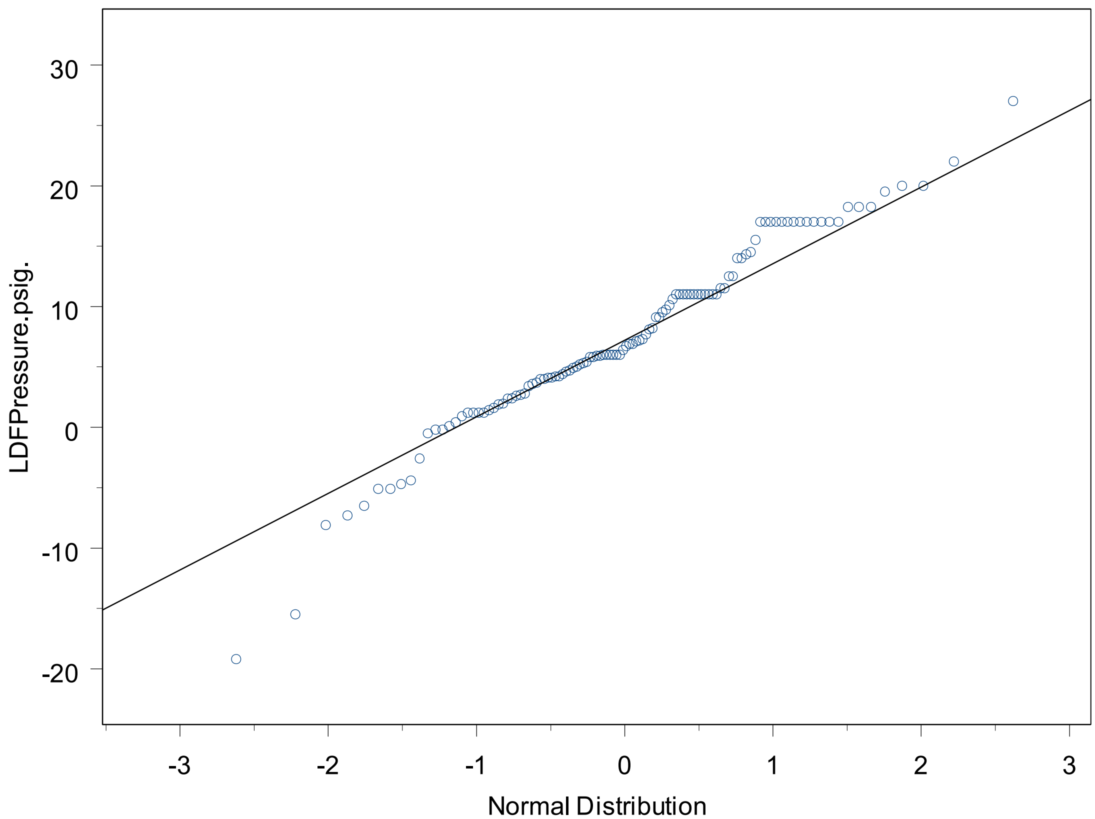
The Hanford data have many more potential outliers (lines with filled circles) than the other sites. These are likely a result of dead zone measurement problems.

Figure 9. Q-Q plots of all LDF psig measurements compare the distribution of these measurements to a normal distribution



The tails of the LDF psig data deviate from the normal line. If the distribution were normal the tails would be closer to the line. These tails are a result of Hanford potential outlier measurements.

Figure 10. Q-Q plot of LDF psig data with Hanford data removed.



The upper tail of these data now approximates a normal distribution. The lower tail has two values that are lower than would be expected. One is from the Innocuous bin, (a Hanford measurement with no dead zone issue), and the other is from SRS. However, it is only the upper tail that is of interest for developing an upper bound on the population and this fit is quite good.