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ProTec[™] TEAR-OFFS: RESULTS OF LONG TERM TESTING

D.K. Peeler

July 2008

Process Science and Engineering Section Savannah River National Laboratory Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500



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REVIEWS AND APPROVALS

AUTHOR:

D.K. Peeler, Process Science and Engineering Section

TECHNICAL REVIEWER:

F.C Raszewski, Process Science and Engineering Section

APPROVERS:

C.C. Herman, Manager, Process Technology Programs

J.C. Griffin, Manager, E&CPT Research Programs

J.E. Occhipinti, Manager, Process Cognizant Engineering Waste Solidification Engineering Date

Date

Date

EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) has completed a series of tests (Phase 1 and Phase 2) to assess the potential use of a Mylar[®] tear-off system as a primary or secondary protective barrier to minimize acid etching ("frosting"), accidental scratching, and/or radiation damage for shielded cells, glovebox, and/or chemical hood windows. Conceptually, thin, multi-layered sheets of Mylar[®] (referred to throughout this report as the ProTec[™] tear-off system) can be directly applied to the shielded cell, glovebox, or hood sash window to serve as a secondary (or primary) barrier. Upon degradation of visual clarity due to accidental scratching, spills/splatters, and/or radiation damage, the outer layer (or sheet) of Mylar[®] could be removed refreshing or restoring the view. Due to the multi-layer aspect, the remaining Mylar[®] layers would provide continued protection for the window from potential reoccurrences (which could be immediate or after some extended time period). Although the concept of using a tear-off system as a protective barrier is conceptually enticing, potential technical issues were identified and addressed as part of this phased study to support implementation of this type of system in the Defense Waste Processing Facility (DWPF). Specific test conditions of interest to the DWPF included the performance of the tear-off system exposed to or under the following conditions:

- (1) acid(s) (concentrated (28.9 M) HF, concentrated (15.9M) HNO₃, 6M HCl, and 0.6M H₃BO₃)
- (2) base (based on handling of radioactive sludges with pH of $\sim 12 13$)
- (3) gamma radiation (due to radioactive sources or materials being used in the analytical cells)
- (4) scratch resistance (simulating accidental scratching with the manipulators), and
- (5) in-situ testing (sample coupons exposed to actual field conditions in DWPF)

The results of the Phase 1 study indicated that the ProTecTM tear-off concept (as a primary or secondary protective barrier) is a potential technical solution to prevent or retard excessive damage that would result from acid etching, base damage (as a result of a sludge spill or splatter), gamma radiation damage, and/or accidental scratching (due to manipulator/tool contact). Although identified as a potential solution, the Phase 1 testing was relatively short-term with exposure times up to 1 - 2 months for the acid and gamma radiation tests. Phase 2 testing included longer exposure times for the acid resistance (up to 456 days) and gamma radiation exposure (700 days with a cumulative gamma dose of ~ 3.1×10^5 rad) assessments. The tear-off system continued to perform well in these longer-term acid resistance testing and gamma exposure conditions. Complete removal of the tear-offs after these long-term exposure times indicate that not only could visual clarity be restored but the mechanical integrity could be retained. The results also provided insight into the ability of the ProTecTM tear-off system to withstand the chemical and physical abuses expected in off-normal shielded cells operations. The conceptual erasing of scratches or marks by excessive manipulator abuse was demonstrated in the SRNL Shielded Cells mock-up facility through the removal of the outer layer tear-off with manipulators.

In addition, the Phase 2 testing included an in-situ assessment of a prototype tear-off system in the DWPF Sampling Cells where the system was exposed to actual field conditions including radioactive sources, acidic and basic environments, dusting, and chemical cleaning solutions over a 5 - 6 month period. DWPF personnel were extremely satisfied with the performance (including the successful removal of 3 layers with manipulators) of the ProTecTM tear-off system under actual field conditions.

The successful removal of the outer layer tear-offs with the manipulator, using tabs not specifically designed for remote operations, demonstrates that the system is "manipulator-friendly" and could be

implemented in a remote environment. The ability to remove the outer layer tear-off not only regains visual clarity but also reduces waste disposal volumes (i.e., disposal of a thin sheet of Mylar[®] which is "collapsible" versus the bulk disposal of a rigid Lexan[®] sheet (alpha shield) or glovebox/sash window) which is more cost effective. The tear-off system could also reduce the number of cell entries needed to replace the Lexan[®] sheet and increase the time interval between glovebox/sash window replacements, which can be costly and time consuming.

Although the primary focus of this study addresses the application of the $ProTec^{TM}$ tear-off system to shielded cells windows, the concept is also potentially applicable to glovebox and hood sash (chemical or radiochemical) windows. In fact, the tear-off concept is potentially applicable to any system where visual clarity is compromised given the environmental conditions of the test. In addition, the tear-offs could be applied to walls or shelves where a protective barrier would reduce deterioration or discoloration.

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LIST OF ABBREVIATIONS

DWPF	Defense Waste Processing Facility
RCT	Recycle Condensate Tank
SRAT	Sludge Receipt Adjustment Tank
SRNL	Savannah River National Laboratory
TRU	Transuranic

1.0 Introduction and Background

The Savannah River National Laboratory (SRNL) has conducted a series tests to assess the use of a thin, multi-layered system of Mylar[®] sheets to serve as a primary or secondary protective barrier against acid etching ("frosting"), accidental scratching (e.g., by manipulator or tool contact), accidental spills/splashes (e.g., sludge and/or acids), and/or radiation/contamination damage (a and/or β) for shielded cells windows. The current mitigation technique used in the SRNL Shielded Cells is to place a rigid, monolithic sheet of Lexan[®] (typically 40" x 36") in front of the glass window as a protective barrier. Over time, visual clarity of the Lexan[®] deteriorates resulting in the need to replace the Lexan[®] sheet about every 1 - 2 years or on an as-needed basis. Replacement is accomplished by cutting a new Lexan[®] sheet to size, retrofitting this sheet with "manipulator friendly" handles, transferring the sheet into the cells, and mounting the sheet into place. Subsequently, the worn Lexan[®] sheet is cut into smaller pieces and disposed of via the appropriate waste disposal route. Similar degradation occurs on glovebox or hood sash windows, but in these cases complete replacement of the window is typically required to regain visual clarity (i.e., generally no primary barrier is used). Whether it is the replacement of a large Lexan[®] sheet in the SRNL Shielded Cells or a direct replacement of a window in a shielded cell, glovebox, or hood sash, replacement and bulk waste disposal are required, which can be time consuming and costly.

As a specific example, in 2003, the Defense Waste Processing Facility (DWPF) replaced alpha shields (similar to the Lexan plates used by SRNL) for seven cell windows due to visual degradation, which obscured the technicians' views and significantly reduced the efficiency of routine tasks. Replacement of the alpha shields required a six week outage in order to decontaminate the cells prior to installation and was estimated to cost \$175K for labor and materials. Within one year of the replacement, visibility of the alpha shields had degraded.

Thin, multi-layered sheets of Mylar[®] (referred to as the ProTec[™] tear-off system) directly applied to the Lexan[®] sheet or alpha shield could result in significant cost reductions through rapid restoration of visual clarity leading to minimal degradation in operator efficiency and overall facility attainment. Upon degradation of visual clarity due to accidental scratching, spills/splatters, and/or radiation damage, the outermost layer of Mylar[®] would be removed to restore the optical clarity. As mentioned, a significant advantage of this system is that it reduces the waste disposal volume (i.e., disposal of a thin sheet of Mylar[®], which is collapsible, versus the bulk disposal of a rigid Lexan[®] sheet or glovebox/sash window that may require size reduction to fit disposal containment). The multi-layer ProTec[™] tear-off system would also reduce the number of cell entries needed to replace the Lexan[®] or alpha shields and increase the time interval between glovebox/sash window replacements which are costly and time consuming.

Although the concept of using a tear-off system as a protective barrier is conceptually enticing, there were potential technical issues identified that needed to be addressed. Based on a literature review of the Mylar[®] product coupled with the potential applications (although customer specific to DWPF), the primary technical issues identified to demonstrate feasibility included:

- > acid/base resistance (e.g., against an acid or sludge spill/splatter)
- scratch resistance (e.g., against accidental contact of the window with the manipulator and/or tool)
- radiation damage resistance (e.g., against radioactive sources in shielded cells and/or gloveboxes)

mechanical integrity after acid/base exposure, scratching, and/or radiation damage (i.e., would the tear-offs lose mechanical strength to the extent that the outer layer tear-off could not be completely removed?).

As reported by George et al. (2005), the results of the Phase 1 testing clearly indicate that the $ProTec^{TM}$ tear-off system concept (as a primary or secondary protective barrier) is a potential technical solution to prevent or retard excessive damage that would result from acid etching, base damage (as a result of a sludge spill or splatter), gamma radiation damage, and/or accidental scratching (due to manipulator/tool contact). Although the Phase 1 results are discussed in detail in that report, a high level overview is provided in the following paragraphs.

With respect to acid resistance testing, no visual or physical degradation was observed on the tear-off systems when exposed to concentrated (28.9M) HF, concentrated (15.9M) HNO₃, 6M HCl, and 0.6M H₃BO₃.^a Although the Phase 1 testing was performed over a relatively short-time period of time (39 days), the test conditions were considered aggressive and included an assessment of an accidental, concentrated HNO₃ spill/splash. The complete removal of the tear-offs after 39 days of exposure (at various heights)^b to all acids suggests that the mechanical integrity of the system was not degraded.

To assess the resistance of the tear-offs to highly basic materials during Phase 1, a simulated DWPF sludge with a starting pH of 12.9 was "splattered" onto the tear-off. The sludge was partially removed using deionized water and a wipe, which left a smear of sludge across both the tear-off system and Lexan[®] blank^c. Upon removal of the outer tear-off, visual clarity was restored on the tear-off system leaving the sludge residue on the Lexan[®] blank. Upon visual inspection of the tear-off, no degradation could be detected. The results indicate that the ProTec[™] tear-off system is also impervious to high pH sample under these test conditions.

The Phase 1 scratch resistance testing demonstrated that marks or scratches can be induced by the manipulators on both the Lexan[®] plate and a tear-off system mounted on a Lexan[®] plate.^d The scratch marks were consistent with those observed in actual service in the SRNL Shielded Cells. Upon removal of the outer layer tear-off with the manipulators, visual clarity was restored. These results confirm the conceptual "erasing" of marks or scratches and the protection potential that the ProTec[™] tear-off system offers. The successful removal of the outer layer tear-off with the manipulator, using tabs (not specifically designed for such a purpose), demonstrates that the system is "manipulator-friendly" and could be implemented in a remote environment.

To assess potential impacts of gamma radiation, a Lexan[®] plate (blank) and Lexan[®] plate with a tearoff system were inserted into Cell #15 in the SRNL Shielded Cells. Cell #15 was selected due to the high gamma background emitted from Co-60 sources (Bibler 2005). Although considered short-term (46 days), the results of the in-situ Cell #15 tests suggest that the concept of the tear-off is feasible under realistic radioactive service conditions. No signs of visual degradation were observed on the tear-offs over the 46 day test period (with a cumulative gamma dose estimated to be ~ 20,000 rad). In addition, the outer layer tear-off was successfully and completely removed suggesting no mechanical degradation after the gamma radiation exposure. The ability of the manipulators to remove the outer

^a The acids and molarities were specified by the DWPF Analytical Laboratory who financially supported this work scope.

^b The Phase 1 tests were initiated with the Lexan blanks and Lexan with tear-offs suspended approximately 4" above the acid baths. After 13 days of testing, the systems were dropped to 2" above the acid vessels where they stayed until Phase 1 was terminated (39 days).

^c Blank indicating that there was no tear-off system mounted on the Lexan plate – Lexan exposed directly to acidic vapors.

^d The use of "scratch resistance" is a misnomer given the Mylar is susceptible to scratching. The purpose of the Mylar is to prevent damage to the cell, glovebox, or sash window to the extent that upon removal of the outer tear-off the scratches or marks would be removed resulting in an unimpeded view.

layer tear-off (using an improvised tab) supported the observations during the mock-up scratch resistance testing. It should be noted that the tear-offs do not provide a protective barrier to the gamma radiation as the penetration depths are too great.

It was recognized that the Phase 1 results were based on relatively short-term exposure times and long-term testing would provide additional insight into the performance and feasibility of this system. The Phase 2 testing not only included longer-term assessments of the acid resistance and gamma radiation impacts, but in-situ service testing in the DWPF Sampling Cells. The focus of this report is to present and discuss the results of the Phase 2 testing of acid resistance, gamma resistance (Cell #15), and DWPF in-situ testing.

2.0 Objectives and Metrics

The objective of the Phase 2 testing is to assess the long-term impacts of acid and gamma radiation exposure on the visual degradation and mechanical integrity of the ProTecTM tear-off system. Long-term exposure to the four acids of interest (concentrated (28.9 M) HF, concentrated (15.9M) HNO₃, 6M HCl, and 0.6M H₃BO₃) covered a time of 456 days. The gamma radiation testing resulted in an exposure time of 700 days (or ~3.1 x 10⁵ rad). These time periods exceed the 1-2 year period in which degradation has typically been observed for both SRNL and DWPF alpha shields or cell windows.

In addition, prototypes of the tear-off system (multiple layers of tear-offs mounted on Lexan[®] plates) were inserted into actual radioactive field conditions within DWPF's Sampling Cells. During the insitu testing, the tear-off systems were exposed to acidic environments (vapor and contact), radioactive backgrounds (as high-level waste was sampled and processed), and manipulator contact over a 5 to 6 month period.

As with the Phase 1 testing, two primary metrics were used to judge if the ProTec[™] tear-off system can be successfully implemented into these hazardous environments:

- Visual clarity
- Mechanical integrity

The latter metric is rather straightforward in terms of classifying success. If the outermost tear-off can not be completely removed by the use of manipulators (or by hand in other applications such as glovebox or hood sash use) after exposure to some environment, then the concept is of no value. Complete removal (i.e. without ripping) of the tear-off after exposure to acids, bases, physical impact, radiation or any combination is of utmost importance.

With respect to visual clarity, the success metric becomes a little more ill-defined. That is, documentation via notebook entries and time-lapsed digital photos provide a visual measure from which to gauge success, but what level of visual degradation would result in a decision to terminate the possible implementation of the concept? The answer may reside not in how much visual degradation occurs, but in how long it takes to reach some critical or detrimental level. Current practice in the SRNL Shielded Cells and DWPF Sampling Cells requires changing of the Lexan[®] sheets or alpha shields on some time interval basis (typically 1 - 2 years). If the tear-offs extend this time interval, one may classify this as a "success." In judging this potential, one also has to remember that the tear-offs are multi-layered so visual degradation success should perhaps be judged on the integrated time the ProTecTM tear-off system provides access to visual clarity relative to the 1-2 year time period typically observed for each Lexan[®] plate.

3.0 Experimental Approach

3.1 Acid Resistance

To support this assessment, four sets of 4 mil (3 layer) $ProTec^{TM}$ tear-offs^e were mounted onto separate Lexan[®] plates and placed on vessels containing four different acids; concentrated (28.9M) HF, concentrated (15.9M) HNO₃, 6M HCl, and 0.6M H₃BO₃.^f In addition to the tear-off system, a blank (Lexan[®] plate without a tear-off system) was also placed on each acid bath as a reference. The Lexan[®] blanks provide a baseline or measure of how aggressive the specific test conditions (acid type and/or acid molarity) were with respect to historical observations in the SRNL Shielded Cells.

The initial outer layer tear-offs were successfully removed on August 8, 2005 after 39 days of exposure at varying heights. This was the termination point for the Phase 1 testing program (see George et al. (2005) for more details). Upon removal, the Lexan[®] blanks and tear-off systems were placed directly on top of the acid bath, referred to as a 0" height set-up,^g which was the initiation point of Phase 2 testing that covered 456 days of exposure. Figure 1 shows the experimental set-up for three of the Phase 2 acid resistance tests. In general, the tear-offs and blanks were inspected on a routine basis for physical degradation and/or visual distortion and photographed periodically for documentation/comparison purposes.^h



Figure 1. Experimental Set-Up for Acid Resistance Testing (0" height).

^e The tear-offs used were obtained from Racing Optics (San Clemente, CA).

^f The acids and molarities were specified by the DWPF Analytical Laboratory who supported this particular work scope. Note in Figure 1, the HNO₃ set-up is not shown as it is located in another hood for safety reasons.

^g Acid levels were approximately ¹/₄" below the top or rim of each vessel – in general no physical contact of acid with the tear-offs occurred

^h All visual and recorded information regarding the Phase 1 and Phase 2 tests are documented in either WSRC-NB-2004-00136 or WSRC-NB-2006-00122.

3.2 SRNL In-Situ Cell #15 Test

The SRNL in-situ cell demonstration was not specifically designed as a bounding assessment, but one that provides insight into the potential use of the ProTecTM tear-off system under realistic test conditions when considering the application in a high radiation environment. Selection of Cell #15 of the SRNL Shielded Cells was primarily based on two factors: the high gamma radiation background (Bibler (2005) reported an average gamma radiation dose of 17.3 rad/hr for Cell #15 due to Co-60 sources being co-located) and the absence of a heat source, which eliminates any possible combustion issues.ⁱ It should be noted that the tear-offs will not be effective in protecting the Lexan[®] plate against gamma radiation (given the high penetration depths), but could be effective in mitigating alpha contamination damage and/or beta radiation damage for other service conditions. However, the gamma radiation may induce embrittlement which may negatively impact mechanical integrity and the ability to completely remove the outer layer tear-off. The in-situ Cell #15 test used a single sheet of Lexan[®] with a 3-layer ProTecTM tear-off system mounted on one side.

Figure 2 is the initial condition of the Lexan[®] sheet prior to Cell #15 entry. Note the use of modified tabs (application of tape in lower right hand corner) on the pre-existing smaller tabs of the tear-off system being used.^j

The Lexan[®] sheet was placed in Cell #15 on July 28, 2005 (

Figure 3) and was monitored for visual clarity on a routine basis. On September 12, 2005 (~46 days of exposure, ~20,000 rad gamma dose) the outer layer tear-off was removed with the manipulators to assess both mechanical integrity and ease of removal with the manipulators.^k Successful removal of this initial outer layer was documented in the Phase 1 report (George et al. (2005)). After the removal of the initial outer layer, the Lexan[®] sheet was placed back on the wall in Cell #15 (with 2 tear-offs remaining) to support the longer term Phase 2 testing. The Phase 2 testing was terminated on June 26, 2007 after approximately 700 days of service with an estimated 310,000 rad gamma dose. During the Phase 2 testing, the 2nd and 3rd layers were removed to assess mechanical integrity and visual clarity.

ⁱ Combustion issues were a concern given the unpredictability of the physical integrity of the tear-offs. More specifically, the tear-off layers are held together by static compression. Assuming that the high radiation background resulted in the separation and free-fall of the sheets, heat sources could cause combustion – an unacceptable situation for the feasibility tests and a service condition or environment that needs to be addressed if implementation is considered.

^j To provide a frame of reference, the Lexan plate is roughly 10" x 10". The tear-offs (mounted on the right hand side) are roughly 9.5" long and \sim 4.5" wide at the maximum width (center or mid-point).

^k Although the removal of the tear-offs does provide insight into feasibility, the size and design of the tear-off tabs should be designed into the fabrication.



Figure 2. Lexan Blank (left) and ProTecTM Tear-Off System (right) Prior to Cell #15 Entry.



Figure 3. Lexan Blank and ProTecTM Tear-Off System in the SRNL Shielded Cells (Day 1).

3.3 DWPF Sampling Cells In-Situ Testing

The in-situ DWPF cell demonstration was initiated to provide additional insight into the potential use of the ProTec[™] tear-off system under realistic test conditions when considering the application in a high radiation environment under prototypic service conditions (not a controlled environment but actual field conditions). On January 12, 2006, DWPF installed one 12" x 12" Lexan[®] plate in Sampling Cell #2 (above the cleaning station) and one 12" x 12" Lexan[®] plate in Sampling Cell #3 (below the Recycle Condensate Tank (RCT) and Sludge Receipt and Adjustment Tank (SRAT) sampling stations). A ProTec[™] tear-off system (3 layers) was mounted onto each Lexan[®] plate prior to cell entry. The Lexan[®] plates with tear-off systems were very similar to those used in the SRNL Cell #15 testing (

Figure 2), including a modification to the pre-existing tab to support manipulator assessments.

Sampling Cell #2 exposed the tear-off system to waste cleaning activities, which involved leaching materials (bottles, wipes, slurries, etc) in 50% nitric acid solution followed by water rinses. Waste cleaning activities are performed to reduce the amount of transuranic (TRU) waste generated. Sampling Cell #3 was selected due to the high volume of sampling of the SRAT product (high pH material) and RCT materials. The materials being sampled and solutions used to clean in both cells are a primary source of window etching and frosting currently experienced in DWPF under current operating conditions. DWPF does have a Lexan[®] alpha shield to help minimize etching or frosting, however, recent experience has indicated that within 1 year of replacement, visual clarity was distorted to the point to which routine operations become less efficient.

The DWPF in-situ testing was performed over a period of approximately 6 months. During the testing, the tear-off systems were exposed to various acidic fumes, radioactive sludge, and various cleaning solutions. The Sample #3 tear-off system was also rinsed with water and acid after each sampling activity or if sludge material came into contact with the coupon. As visibility became an issue, the ProTec[™] tear-offs were visually evaluated and the outer layer was removed using manipulators. Only visual observations and direct feedback from the DWPF Sampling Cell personnel were obtained during the 5-6 month testing.

4.0 Results

4.1 Acid Resistance at 0" Height

Table 1 summarizes the major events in chronological sequence for both phases of the acid resistance testing. Along with the major events, high level summary comments are also provided. The table is provided as a reference guide for the upcoming discussions.

The 0" height tests were initiated on August 8, 2005 just after the successful removal of the initial tear-off layer (1st of three). After 35 days of Phase 2 testing, the only signs of visual degradation were observed on the Lexan[®] blank for concentrated HNO₃ (Figure 4). No optical distortion was observed on any of the tear-offs after 35 days at the 0" height. These results indicate that the ProTecTM tear-offs would provide more protection (in terms of time exposure) as compared to the Lexan[®] blank (or plate) under this specific test condition. It should be noted that the HNO₃ fumes or vapors appear to have caused some reaction with the glue holding the ProTecTM sheet to the Lexan[®] plate (right hand side of Figure 4). Although some reaction (or staining) of the glue is noted, the ProTecTM sheets remained adhered to the Lexan[®] plate throughout the Phase 2 testing (456 days of exposure).

Event/Observations	Date	Comment
Initiated 0" height acid resistance	August 8, 2005	Initial tear-off layers were successfully removed
testing for Lexan [®] blanks and tear-		for all acid tests on August 8, 2005. ¹ The 0" level
offs		represents essentially no physical gap between the
		action bath and the blank of tear-oil. This is an
		conditions.
Yellow circular stain noted on Lexan [®]	August 25, 2005	After 17 days at the 0" level, no signs of "yellow"
blank for the HNO ₃ acid test		circular stain on tear-off system for HNO ₃ , all
		other Lexan [®] blanks were visually clear.
Completion of Phase 1 Acid	September 12, 2005	35 days total exposure at 0" height (2 nd layer tear-
Resistance Testing		off), no visual signs of degradation, "yellow" stain
(as reported in WSRC-1R-2005-		on Lexan [®] blank (HNO ₃) progressing, outer layer
00386)		tear-offs were not removed.
No reaction on Mylar [®] tear-offs,	November 30, 2005	126 days total exposure at 0" height (2 nd tear-off),
yellow stain on HNO3 Lexan [®] blank		no visual signs of degradation on tear-offs, outer
progressing		layer tear-offs were not removed.
Possible yellow stain on tear-off	June 20, 2006	314 days at 0" height, possible yellow stain on
exposed to HNO ₃		HNO ₃ tear-off system, stain on Lexan [®] blank
		progressing
Yellow stain on tear-off exposed to	September 28, 2006	414 days total exposure at 0" height for 2 nd tear-
HNO ₃		off, no visual signs of degradation on tear-offs
		with exception of HNO ₃ bath. Yellow stain
		potentially due to liquid contact – overfilling of
		HNO ₃ acid bath. Outer layer tear-offs were not
Removal of 2 nd layor (1 st layor after	November 0, 2006	156 days total experience at 0" height no signs of
exposure at 0 " height)	November 9, 2000	visual degradation of tear-offs with exception of
exposure at o height)		HNO_{2} tear-offs were successfully removed from
		concentrated (28 9M) HF 6M HCl and 0 6M
Termination of Acid Resistance		H_3BO_3 systems. The tear-off exposure (immersed)
Testing		in concentrated (15.9M) HNO ₃ , did tear when
		removal was attempted.

Table 1. Summary of Acid Resistance Testing at 0	Table 1.	Summary	of Acid I	Resistance	Testing at 0'
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On November 30, 2005 (after 126 days of exposure at 0"), visual observations of each system indicated no change in terms of visual degradation on all of the tear-off systems. With the exception of the HNO₃ test, no reaction or visual degradation was observed on the Lexan[®] blanks. The Lexan[®] blank exposed to the HNO₃ did show signs of an increasing progression of visual distortion due to the yellow haze.

On June 20, 2006 (after 314 days of exposure at 0"), a slight yellow stain was observed on the tear-off system exposed to HNO_3 . This was the first indication of any type of reaction leading to some degree of visual distortion on any of the ProTecTM tear-offs. Although visual clarity had been degraded, a decision was made not to remove the outer layer tear-offs (the 2nd of three layers) on any of the systems. After 414 days of service (visual observations noted on September 28, 2006), no visual signs of a reaction or visual distortion was noted on the concentrated (28.9M) HF, 6M HCl, and 0.6M

¹ During termination of the Phase 1 tests, two layers were accidentally removed from the 6M HCl system; leaving only one layer remaining on this system.

 H_3BO_3 systems (either Lexan[®] blanks or tear-offs). Figures 5-7 show the visual clarity of the HF, HCl, and H_3BO_3 systems, respectively.

Figure 8 shows the yellow stains or reactions on both the Lexan[®] plate and tear-off exposed to the HNO_3 bath after 414 days. It was noted that during the filling of the HNO_3 bath after 314 days of exposure (but prior to the 414 days of exposure), the vessel was apparently overfilled leading to physical contact of the HNO_3 with the $ProTec^{TM}$ tear-off which increased the rate of attack on the tear-off. Physical property data of $Mylar^{®}$ suggests that immersion in HNO_3 leads to a significant reduction in mechanical strength.^m

On November 9, 2006, after 456 days of exposure, the Phase 2 testing was terminated. Visual observations were recorded and the 2^{nd} tear-off was removed to assess mechanical integrity. As with previous visual observations, no visual distortion was noted on the concentrated (28.9M) HF, 6M HCl, and 0.6M H₃BO₃ systems (either Lexan[®] blanks or tear-offs) as shown in Figures 9-11. However, the yellow stains as a result of the HNO₃ exposure or contact were still apparent on both the Lexan[®] plate and tear-off as shown in Figure 12.

After 456 days of exposure to the various acids, a decision was made to remove the outer tear-off from each system. As with the previous removal (August 8, 2005), the outer layer tear-offs for the concentrated (28.9M) HF, 6M HCl, and 0.6M H₃BO₃ systems were easily removed in their entirety indicating that mechanical integrity was not compromised. Figures 13-16 show a series of photos prior to, during, and after the removal of the second tear-off from the concentrated (28.9M) HF, 6M HCl, and 0.6M H₃BO₃ systems. All of the before, during, and after photos for each specific system are not shown, but similar results were obtained. Figure 13 shows the pre-removal condition of the 6M HCl tear-off system.ⁿ In this figure "rings" are observed where the tear-off system rested on the HCl vessel for the 456 days. The rings could be a result of a reaction of the tear-off with the HCl and/or surface roughness (small scratches) from contact with the plastic acid vessel. Independent of the source, when the tear-off for this system). The "rings" were removed via removal of the tear-off, restoring visual clarity, which is the general objective of the system. Figure 14 shows the last tear-off being removed from the 6M HCl system. It should be noted that no residual glue on the Lexan[®] plate was observed upon complete removal of this layer.

Figure 15 and Figure 16 capture the removal of the 2^{nd} tear-off and post-removal condition for the concentrated HF system. As with the 6M HCl and 0.6M H₃BO₃ systems^o, complete removal of the outer-layer tear-offs was achieved. These results suggest that the ProTecTM tear-offs are a viable system capable of withstanding relatively aggressive vapor attack from these specific acids, while still offering the opportunity to restore visual clarity when needed. The fact that the 2^{nd} layer tear-offs were exposed for 456 days suggests that (at a minimum) the Lexan[®] sheets currently used by the

^m Technical information (albeit limited) was obtained on a DuPont Mylar product similar to the multi-layer tear-off product to be used in this testing. The technical data indicated that the chemical, electrical, optical, and physical – thermal properties of Mylar vary as a function of test or environmental service conditions. For example, mechanical degradation data (e.g., tensile strength) were presented for various acid molarities (including HCl and HNO₃) and exposure times. In general the data suggested that as the acid molarity increased, the loss of mechanical strength increased (which could have an impact on the ability to remove the outer layer tear-off). The data associated with optical integrity suggested that "hazing" occurred upon exposure to various acids and bases. Although degradation of mechanical integrity and/or optical quality was noted, the standard testing performed to obtain this information was based on immersion tests – not typical (not even an extreme condition) for the planned application.

ⁿ As previously mentioned, on August 8, 2005, an additional tear-off (2 in total) was unintentionally removed from the 6M HCl system leaving only one ProTec[™] tear-off.

 $^{^{\}circ}$ For documentation of the removal of the outer layer tear-off for the 0.6M H₃BO₃ system, refer to pages 32 – 33 of WSRC-NB-2006-00122.

SRNL Shielded Cells (which are replaced every 1-2 years) could have an increased service life (3-4 times) based on the multi-layer ProTec[™] tear-off concept.

With respect to the HNO₃ test, an attempt was made to remove the outer layer tear-off from this system to determine if the "yellow stain" could be removed leaving a fresh (and final) tear-off on the Lexan[®] plate after 456 days of exposure. Figure 17 shows the tear-off system exposed to HNO₃ prior to removal. Removal of the outer layer tear-off resulted in fracture or tearing (i.e., incomplete removal of the tear-off). In fact, when attempts to remove the outer layer were initiated, it was determined that the two remaining layers had "fused" together. These results were discouraging from the standpoint of not having a long term data point in which complete removal was successful. In hindsight, once the yellow stain had been observed (at the 314 day mark) or shortly after, the outer (2nd) layer should have been removed to gain insight into mechanical integrity issues and to evaluate the potential to remove the stain. If not, then the decision to remove the outer layer (2nd tear-off) should have been made once physical contact between the HNO₃ and the tear-off was noticed (at the 414 day mark). The tear-offs were potentially in direct contact with HNO₃ for 30-40 days, which may be considered a condition under which this system would (and should) not be utilized.



Figure 4. Visual clarity of Blank (left) and Lexan[®] + Tear-Off (right) after 35 days at the 0" mark (concentrated HNO₃). Note: Yellow stain on Blank and vapor attack on glue holding the tear-off to Lexan Plate.



Figure 5. Visual clarity of Blank (left) and Lexan[®] + Tear-Off (right) after 414 days (September 28, 2006) at the 0" mark (concentrated HF).



Figure 6. Visual clarity of Blank (left) and Lexan[®] + Tear-Off (right) after 414 days (September 28, 2006) at the 0" mark (6M HCl). Note: the outer layer is the third and final layer on this system.



Figure 7. Visual clarity of Blank (left) and Lexan[®] + Tear-Off (right) after 414 days (September 28, 2006) at the 0" mark (0.6M H₃BO₃).



Figure 8. Yellow stains on both the Blank (left) and Lexan[®] + Tear-Off (right) after 414 days (September 28, 2006) at the 0" mark (concentrated HNO₃).



Figure 9. Visual clarity of Blank (left) and Lexan[®] + Tear-Off (right) after 456 days (November 9, 2006) at the 0" mark (concentrated HF).



Figure 10. Visual clarity of Blank (left) and Lexan[®] + Tear-Off (right) after 456 days (November 9, 2006) at the 0" mark (0.6M H₃BO₃).



Figure 11. Visual clarity of Blank and Lexan[®] + Tear-Off (right) after 456 days (November 9, 2006) at the 0" mark (6M HCl). Note: the outer layer is the third and final layer on this system.



Figure 12. Yellow stains on both the Blank (left) and Lexan[®] + Tear-Off (right) after 456 days (November 9, 2006) at the 0" mark (concentrated HNO₃.



Figure 13. Pre-removal condition of the 6M HCl ProTec[™] Tear-Off system (456 days at 0").



Figure 14. Final ProTecTM Tear-Off being removed from the 6M HCl system (456 days at 0"). Note: no residual "glue" left on the Lexan[®] plate.



Figure 15. Final ProTec[™] Tear-Off being removed from the concentrated HF system (456 days at 0").



Figure 16. Post-removal of the outer ProTec[™] Tear-Off from the concentrated HF system (456 days at 0").



Figure 17. Pre-removal condition of the concentrated HNO₃ Tear-Off system (456 at 0").

4.2 In-Situ SRNL Shielded Cell (Cell #15) Testing

Table 2 summarizes the major events in chronological sequence for SRNL Cell #15 gamma radiation testing. Along with the major events, high level summary comments are also provided. The table is provided as a reference guide for the upcoming discussions.

Event/Observations	Date	Comments
Lexan [®] tear-off system placed in SRNL Shielded Cells (Cell #15)	July 28, 2005	Initiation of Gamma Radiation testing (see Figure 18)
Completion of Phase 1 In-Situ Cell #15 Testing, 1 st outer layer tear-off successfully removed (see Figure 19)	September 12, 2005	46 days of service, ~20,000 rad dose, no visual signs of degradation, complete removal of tear-off with manipulator.
Visual Observation	December 12, 2005	91 days of service, no visual signs of degradation, outer layer tear-off not removed
2 nd layer removed (see Figure 20)	June 20, 2006	328 days of service, ~155,000 rad gamma dose, no visual signs of degradation, outer layer tear-off successfully removed, "hazing" observed on Lexan [®] plate
Visual Observation	September 26, 2006	426 days of service, ~205,000 rad dose, no visual signs of degradation, outer layer not removed
Removal of 3 rd (and final) tear-off layer (see Figures 21- 23) (Termination of Phase 2 Testing)	June 26, 2007	~700 days of service, 300,000 rad dose, no visual signs of degradation, outer layer (final) tear-off completely removed, isolated spots
		of glue residue noticed on Lexan [®] plate

 Table 2. Summary of Gamma Radiation Testing (Cell #15)
 Image: Cell #15

Figure 18 shows the "blank" and "tear-off" systems hanging from an intermediate wall in Cell #15 just after cell entry (July 28, 2005). The "tear-off" is visible on the right hand side of the Lexan[®] plate, which is aligned vertically. As previously mentioned, the average radiation dose in Cell #15 is 17.3 rad/hr (Bibler (2005)). The SRNL Shielded Cells personnel monitored the system on a daily basis for any signs of visual distortion or mechanical degradation.

As documented in WSRC-TR-2005-00386, after 46 days of exposure (estimated cumulative gamma dose of \sim 20,000 rad), there were no signs of visual distortion as shown in Figure 19 (photo taken prior to the removal of the outer layer tear-off on September 12, 2005). After documenting the visual clarity of the system, the SRNL Shielded Cell technician used the manipulators to remove the outer layer tear-off. The outer layer tear-off was completely removed (i.e., no ripping which demonstrated some degree of mechanical integrity after a cumulative dose of \sim 20,000 rad of gamma exposure) and left the two remaining tear-offs intact and undamaged.



Figure 18. Lexan[®] plate with Tear-Off system (right) introduced into Cell #15 on July 28, 2005 (day 1).



Figure 19. Lexan[®] plate with Tear-Off system in the SRNL Shielded Cells prior to removing the outer Tear-Off layer (day 46 – termination point of Phase 1 testing).

On June 20, 2006, the second tear-off was removed after 328 days of exposure (~155,000 rad). Prior to removal of the outer layer (the 2nd of three layers), no visual degradation was noted. The outer layer was easily and completely removed (i.e. without ripping) with the manipulators, which is consistent with the initial removal on Day #46 (September 12, 2005). The successful removal provided sound technical data to suggest that the tear-off could withstand up to approximately 155,000 rad (gamma) without losing mechanical integrity. After removal of the second layer, there was a noticeable distinction between the "fresh" tear-off and the Lexan[®] blank in terms of visual clarity. Figure 20 shows the system just after removal of the outer layer tear-off. On the right hand side, the reflection of the pipettes or small sample bottles is relatively sharp as compared to the more diffuse Lexan[®] blank side. This result suggests that some type of residue was coating the entire system (not evident prior to removal) and upon removal of the tear-off, the deposited layer was removed.



Figure 20. Post-removal of the outer ProTecTM Tear-Off layer after 328 days of exposure (~155,000 rad).

Visual observations were made on September 26, 2006 and January 1, 2007 with no signs of visual distortions or mechanical degradation. On June 26, 2007, after approximately 700 days of service and an estimated 330,000 rad exposure, the third and final tear-off was removed. Prior to removal, there were no visual signs of degradation (see Figure 21). Although the final tear-off was successfully removed (i.e., one single sheet), there was an increased resistance to removal. In fact, after removal, some residual glue was observed on the Lexan[®] plate in isolated spots (see Figures 22 and 23).^p In terms of possible impacts for certain applications, the presence of residual glue may result in the need for removal prior to applying a second series of tear-offs to the Lexan[®] plate. In other situations (i.e., where replacement of the entire Lexan[®] sheet being used as an alpha shield will be made), the glue may not pose any significant issues as visual clarity in the majority (central

^p As noted in Section 4.1, removal of the final tear-off from the HNO₃ acid resistance testing showed no sign of residual glue remaining on the Lexan plate.

portion) of the Lexan[®] plate would allow operations to continue. If the glue backing were to present a possible issue with specific applications, research could be performed to develop an alternative adhesive that would maintain integrity during use but minimize or eliminate the residue upon removal. Again, the tear-off systems being used in this testing are "off-the-shelf" systems, which are not specifically designed for the applications being tested (although effective).



Figure 21. Pre-removal of the outer Tear-Off layer after approximately 700 days of exposure (~330,000 rad) on June 26, 2007.



Figure 22. Post-removal of outer Tear-Off layer after approximately 700 days of exposure (~330,000 rad) on June 26, 2007. Note: residual glue is located near the center of the Lexan[®] plate.



Figure 23. Post-removal of the outer Tear-Off layer after approximately 700 days of exposure (~330,000 rad) on June 26, 2007. Note: residual glue located near the center of the Lexan[®] plate.

4.3 In-Situ Testing in DWPF Sampling Cells

Table 3 summarizes the major events in chronological sequence for the in-situ DWPF Sampling Cells testing program. Along with the major events, high level summary comments are also provided. The table is provided as a reference guide for the upcoming discussions.

Event/Observations	Date	Comments
Two tear-off systems placed in DWPF	January 12, 2006	One tear-off system placed in Cell
Analytical Cells		#2 above cleaning basket. One tear-
		off system placed in Cell#3 below
		RCT and SRAT sampling station.
Removal of 1 st tear-off from both	April 26, 2006	105 days of service, complete
systems		removal of tear-offs with
		manipulator.
		(Ease of removal consistent with
		SRNL Cell #15 testing)
Removal of 2 nd and 3 rd layers not	Not recorded	Not recorded
documented		
Memorandum received from Janice	June 6, 2006	Approximately 6 months of service
Cook on performance of tear-offs in		
DWPF analytical cells		

 Table 3. Summary of in-situ DWPF Sampling Cells testing

On April, 26, 2006 (105 days of service), SRNL personnel witnessed the removal of the initial outer layer tear-offs from both systems using manipulators. The remote removal of the outer layers appeared quite easy and was consistent with the removal of the initial layers in the SRNL Shielded Cell testing (see Section 4.2). The removal of the 2nd and 3rd layers were not formally documented by DWPF analytical personnel or communicated to SRNL. However, on June 9, 2006 (after approximately 6 months of in-situ service), Cook (2006) issued an internal memorandum documenting the test conditions and the results. Excerpts from the memorandum include:

"Removal of the ProTecTM sheets from the coupons with the manipulators proved to be achievable with the installation of the larger tabs. The Mylar[®] layer was able to be removed effortlessly with the manipulators."^q

Additional feedback from DWPF personnel stated:

"Due to the cell environment with the sludge material, the (unprotected) Lexan[®] sheet visibility was ineffective due to fine powder residue that coats everything in the cells. Rinsing with water or 50% nitric acid after sampling activities did not remove the fine powder. If sludge material came into contact with the Lexan[®] itself, a stain ring would remain on the Lexan[®] after rinsing."

The conclusions drawn from DWPF personnel indicated that:

^q As with the SRNL Cell #15 testing (

Figure 2), the pre-existing tabs were modified to support the ability of the manipulators to grasp each individual tear-off.

"The ProTec[™] sheet will prolong the lifetime of the alpha shields and provide good visibility for personnel. Use of (unprotected) Lexan[®] plates to protect the alpha shields proved to be ineffective due to the corrosive environment and the dry powder residue."

5.0 Summary

With respect to acid resistance testing, no visual or physical degradation was observed on the tear-off systems when exposed to concentrated HF, concentrated HNO₃, 6M HCl, and 0.6M H₃BO₃ through a testing period of 456 days. The test conditions are considered "aggressive" as the tear-offs were directly exposed to the various acid vapors and included an assessment of an accidental HNO₃ spill (Phase 1 testing). The observation of a circular yellow stain on the Lexan[®] blank under HNO₃ acid exposure at 0" after 17 days, clearly indicates that the ProTecTM tear-offs may be more chemically resistant than the Lexan[®] sheet under those service conditions. The complete removal of the tear-offs after long-term exposure (at a 0" height) suggests that the mechanical integrity of the ProTecTM system was not degraded.

To assess the resistance of the tear-offs to highly basic materials, a simulated DWPF sludge with a starting pH of 12.9 was "splattered" onto the tear-off. The sludge was partially removed through the use of deionized water and a wipe leaving a smear of sludge across both the tear-off and Lexan[®] "blank". Upon removal of the outer tear-off, visual clarity was restored to the tear-off portion leaving the sludge residue on the Lexan[®] blank. Upon visual inspection of the tear-off, no visual sign of reaction or degradation could be detected. The results indicate that the ProTecTM tear-off is impervious to the high pH sample.

The "scratch resistance" testing demonstrated that marks or scratches can be induced by the manipulators (in "mock-up") on both the blank and tear-off systems. The scratch marks were consistent with those observed in actual service in the SRNL Shielded Cells. Upon removing the outer tear-off layer with the manipulators, visual clarity was restored. The results of this test not only confirms the conceptual "erasing" of marks or scratches, but also demonstrates the ability of the manipulators to grasp and remove the outer layer tear-off.

The results of the in-situ Cell #15 tests suggest that the concept of the tear-off is feasible under realistic service conditions as measured by high gamma radiation. The tear-offs showed no signs of visual degradation up to 700 days exposure (with a cumulative gamma dose estimated to be \sim 330,000 rad). In addition, the outer layer tear-off was successfully and completely removed suggesting no mechanical degradation after the gamma radiation exposure. The ability to remove the outer layer tear-off (using an "improvised" tab) supported the observations during the "mock-up" scratch resistance testing that the manipulators can effectively remove the outer layer. It should be noted that the tear-offs do not provide a protective barrier to the gamma radiation as the penetration depths are too great. The outer layer tear-off would provide a protective barrier against α and/or β radiation (conditions recommended for further testing are discussed in Section 6.0).

The results of the Phase 1 and Phase 2 studies clearly indicate that the ProTec[™] tear-off concept is a potential technical solution to mitigate excessive damage that would result from acid etching or spills, base damage (as a result of a sludge spill or splatter), gamma radiation damage, and/or accidental scratching (due to manipulator/tool contact). The tests performed in this task showed that ProTec[™] tear-offs can withstand the chemical and physical abuses expected in abnormal shielded cells operations. The tear-offs not only provide some measure of acid resistance, as reflected by the lack of

visual degradation after being exposed to four acids, but also act as a protective barrier to accidental contact with the manipulators and/or tools. The conceptual "erasing" of scratches or marks was demonstrated in the SRNL Shielded Cells mock-up facility through the removal of the outer layer tear-off with manipulators.

The successful removal of the outer layer tear-off with the manipulator, using tabs not specifically designed for such a purpose, demonstrates that the system is "manipulator-friendly" and could be implemented in a remote environment. The ability to remove the outer layer tear-off not only regains visual clarity but also reduces waste disposal volumes (i.e., disposal of a thin sheet of Mylar[®] which is "collapsible" versus the bulk disposal of a rigid Lexan[®] sheet or glovebox/sash window) which is more cost effective. The "tear-off" system could also reduce the number of cell entries needed to replace the Lexan[®] sheet and increase the time interval between glovebox/sash window change outs which can be costly and time consuming.

6.0 Recommendations

The following recommendations are made to advance the potential use of the ProTec[™] tear-offs as a primary or secondary protective barrier. Prior to implementation, the following items should be considered:

- Identify other environmental conditions (e.g., formic acid, NaOH, sodium peroxide) in which the tear-off system may be implemented and perform feasibility or scoping studies to address resistance issues.
- Obtain additional data on gamma radiation effects using a Co-60 source and potential interactive effects (such as radiation damage coupled with acid exposure) on visual clarity and mechanical integrity.
- Obtain data on alpha and/or beta radiation damage/contamination to the tear-offs. This may be of particular interest for glovebox usage (e.g., actinide bearing materials).
- Perform assessments on thermal stability / fire resistance in case service conditions call for elevated temperatures. DuPont states that the melting point of their Mylar[®] film is approximately 250°C. If service conditions approach or exceed this value, then the application of Mylar[®] tear-offs may be limited.

7.0 License and Patent Application

Premier Technology, Inc. (Blackfoot, Idaho) has obtained exclusive licensing rights for the ProTec[™] tear-off system from the Washington Savannah River Company (WSRC).^r

8.0 Acknowledgements

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^r WSRC Licensing Agreement No. LA-07-008.

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