APPLICATION NOTE

Liquid Scintillation

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Improve Results Obtained from Swipe Assays

Introduction

Swipe assays are routinely performed in laboratories and other facilities that use or handle radioactive materials. These assays, often referred to as smear or wipe tests, are performed to comply with radioactive material license requirements, assure laboratory safety, and provide information that proper handling procedures are being followed. Typically, swipes are performed to monitor for the presence of removable surface contamination from low energy β–emitting radionuclides such as ^{3}H, ^{14}C and ^{35}S. Swipe assays are also used to detect the presence of α–contamination.

Although the merit of relying on swipe tests for detecting removable contamination has been questioned in a publication by Klein et al.,[1] this method remains a universally accepted technique. In fact, it is often a stipulation of a radioactive material possession license. The U. S. Nuclear Regulatory Commission (NRC, 1981) suggested that 100 cm^2 areas be wiped and lists acceptable levels for surface contamination[2]) (22000 DPM/100 cm^2 equivalent to 367 Bq/100 cm^2) in restricted areas. Furthermore, there is no practical alternative to monitor for the presence of weak β–emitters, especially tritium, than by swipe testing followed by liquid scintillation counting (LSC).

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Admittedly, there can be considerable variability in the results obtained from swipe tests due to the types of surfaces monitored, the type of swipe material used, and the counting efficiency of the radioactive material deposited on the swipe. However, these tests provide some measure of the amount of removable contamination present.

A number of investigators including Klein et al, Kobayashi, Takiue et al, and others have performed studies to evaluate the collection and counting efficiency of various swipe materials and methods for the detection of both β– and α–radionuclide contamination[1,3,4,5]. Both collection and counting efficiency influence the amount of contamination that can be detected. This application note will review their findings and offer suggestions to improve the results from swipe assays.

Experimental Methods – A Historical Overview

Experiments have been performed to test the applicability of various types of materials and techniques for doing swipe tests. Often, the materials routinely chosen to perform these tests are those that are readily available in the laboratory. Many types of swipe materials have been tried including paper, styrofoam, cotton swabs, cloth patches, and glass fiber filters.

Swipe Tests for β–Activity: Collection Efficiency

Two common collection devices, cotton swabs and 2.5 cm diameter glass fibre filter disks, were used in experiments performed by Klein et al. They investigated collection efficiencies using dry wipes as well as those dampened with different amounts of distilled water, 70% ethanol, or a working strength of a multipurpose laboratory glassware detergent.

In this study, clean unwaxed surfaces, representative of laboratory spaces (such as vinyl floor tile, plate glass, and fresh lead foil), were marked with a 5.1 cm * 5.1 cm grid pattern. Aliquots of a known amount of either ^{14}C-glucose or ^{32}P-guanidine triphosphate were spotted and dried in the middle of each marked area. The entire area of each square was wiped with a circular, inward motion with consistent force. Three to five replicate squares were sampled for each combination of detection device and surface type. The samples were counted for one minute on a PerkinElmer TriCarb 2000CA LSC (similar to B2810, B2910, B3110) in 7 ml standard glass vials containing 6 ml of a universal type cocktail. Counting efficiencies of the method were determined by pipetting 0.1 ml of the ^{14}C- or ^{32}P-source material on the swabs or glass fiber disks in triplicate and counting the samples as described above. These internal standards were used to correct counting efficiencies to 100% so that the wipe testing conditions could be directly compared.

Figure 1 shows the collection efficiency obtained with either dry or pre-wetted (with about 75 µl of H_2O) wipes from 25 cm^2 squares of vinyl floor tile, plate glass, and lead foil. Collection efficiency varied with both the wipe method and the surface wiped. In most cases, collection efficiencies are enhanced by at least a factor of two after dampening either the swabs or the filter disks with water.

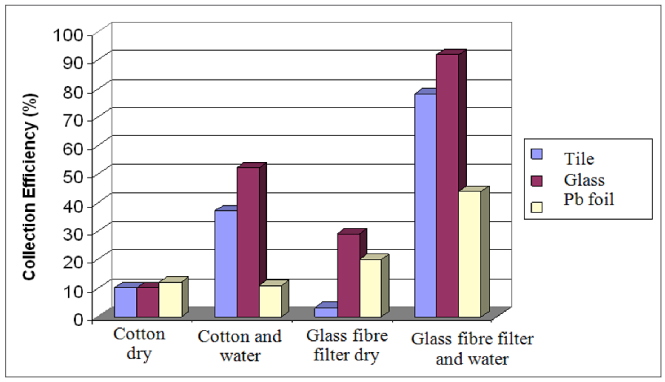

Figure 1. Collection efficiency for ^{32}P-GTP wiped from 25 cm^2 squares. Data from Klein et al.

Klein et al reported[1] that dampening with ethanol or a lab detergent produced results that were statistically indistinguishable from swipe devices dampened with an equal volume of water. The author also reported that collection efficiency is dependent on the volume of liquid added to the wiping device, with 20-100 µl providing the highest efficiency. The overall conclusion of this study is that the glass fiber filter disks appear to provide consistently higher collection and counting efficiencies, but cotton swabs offer flexibility, speed, and reduced handling because they are convenient to use and can be easily placed in a counting vial after the swipe is taken. This reduced handling will minimize the spread of contamination to other swipes or personnel. Similar experiments were performed at the U. S. Department of Energy Battelle Pantex plant in Texas with ^{3}H-glucose[5]. In this experiment, a known activity (100 µl containing 5800 DPM) of ^{3}H-glucose was pipetted and dried in the center of the squares of a clean foil-lined cardboard grid. The collection efficiencies of cotton swabs, paper disks, and foam squares were tested. The same activity was added directly to the cocktail (Ultima Gold™) which served as an internal standard for recovery calculations. The results of this study are summarized in Figure 2.

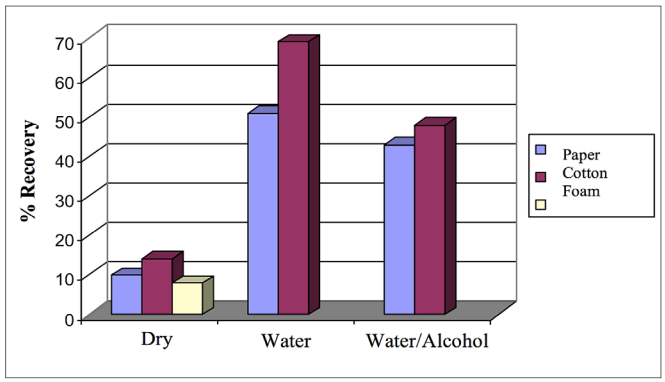

Figure 2. Collection efficiency of Tritium surface monitoring (wiped from foil)). Note: Foam swipes were not treated with water or water/alcohol. Data from Battelle Pantex.

In all cases, pre-wetting the collection device with approximately 100 µl of wetting agent resulted in a dramatic improvement in collection efficiency.

In another study, Takiue et al proposed the use of a water-soluble paper (water-soluble tack paper, Tomoegawa Paper Company) for swiping both α– and β–emitters followed by liquid scintillation counting[4]. Water soluble paper was proposed as the test swipe device since it can be homogeneously dispersed into a liquid scintillator by wetting the paper with a small amount of water before adding cocktail. Collection efficiencies from a polyvinyl chloride surface and aluminium plate obtained with this paper are comparable to using conventional smear paper. The type of conventional smear paper used was not discussed. The authors report a 63.0 +/- 1.6% vs. 64.6 +/- 1.9%, collection efficiency from the polyvinyl chloride plate for the water-soluble paper and the conventional smear paper, respectively. Collection efficiencies of 27.2 +/- 1.6% und 30.6 +/- 1.6%, respectively, were obtained for both devices from the aluminium plate. The advantage of ensuring that the sample is homogeneously dispersed in the cocktail is obvious by comparing the pulse height distributions obtained from conventional smear paper, water-soluble paper, and a homogeneous sample. This comparison is shown in Figure 3 for ^{3}H swipe samples. The lower pulse height for ^{3}H on the conventional smear paper is the result of β–particle self absorption and photon reduction inside the paper. The authors conclude that good agreement between the water-soluble paper sample and the homogeneous sample with the same degree of quench will improve the accuracy of determining the total activity on the swipe.

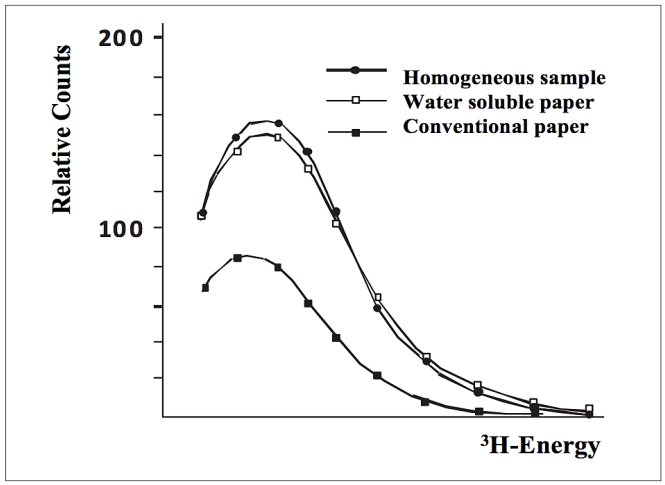

Figure 3. Pulse height distributions of ^{3}H smear samples. Data from Takiue et al.

Swipe Tests for β–Activity: Counting Efficiency

In a recent study by Kobayashi, four swipe media (filter paper, glass fiber, cotton swab, and Styrofoam) were evaluated for their ability to release water-soluble radiolabeled compounds into five cocktail solutions containing two percent water[3]. The radiolabeled compounds were ^{3}H-leucine, ^{14}C-glycine, and ^{32}P-ATP. The cocktails were Insta-Gel XF, Pico-Fluor LLT, Hionic-Fluor, Ultima Gold XR, and Opti-Fluor from PerkinElmer. For a more detailed description of cocktails and their properties please read application note 16[8]. This study did not investigate the collection efficiency of the various swipe devices, but focused on the efficacy of recovering activity from the four wipe media. A known amount of activity in a 10 µl volume was pipetted onto the surface of each wipe medium and allowed to dry overnight at room temperature. All the samples were assayed in triplicate. The dried swipes were placed in standard 20 ml glass LSC vials containing 10 ml of cocktail loaded with two percent water and counted in a TriCarb 2550TR/LL LSC (comparable to current B3110/LL). This instrument is comparable with the TriCarb models B2910TR or B3110TR with ultra low level option. Because water is part of the counting solution and the radiolabeled compounds are water soluble, good recoveries were obtained for the radionuclides when counted immediately. Recovery improved significantly for ^{3}H and ^{14}C after 48 hours. The effect is shown in Tables 1 and 2, respectively.

Table 1. Recovery and counting efficiencies of ^{3}H-contaminations.*

Cocktail	Efficiency (%)	Recovery (%)			
		Paper	Glass Fiber	Cotton Swab	Styrofoam
Ultima Gold XR	48	79 (58)	95 (93)	72 (56)	95 (88)
Insta-Gel XF	57	59 (47)	92 (89)	67 (53)	76 (70)
Pico-Fluor LLT	54	84 (56)	100 (100)	90 (61)	100 (100)
Hionic-Fluor	51	83 (53)	100 (98)	84 (57)	100 (100)
Opti-Fluor	20	73 (49)	89 (84)	73 (54)	83 (77)

* 2260 +/- 156 DPM ^{3}H-leucine were measured in triplicates and in 10 ml counting solution containing 2% water. The samples were counted immediately, after 24 hours, and after 48 hours.

Table 2. Recovery and counting efficiencies of ^{14}C-contaminations.†

Cocktail	Efficiency (%)	Recovery (%)			
		Paper	Glass Fiber	Cotton Swab	Styrofoam
Ultima Gold XR	95	92 (73)	100 (100)	92 (85)	96 (100)
Insta-Gel XF	96	82 (78)	100 (100)	87 (85)	100 (100)
Pico-Fluor LLT	96	97 (77)	100 (100)	97 (84)	100 (100)
Hionic-Fluor	96	89 (76)	100 (100)	92 (83)	100 (97)
Opti-Fluor	88	92 (76)	99 (99)	88 (76)	96 (97)

† 9050 +/- 347 DPM ^{14}C-glycine were counted in triplicates and counted in 10 ml counting solution containing 2% of water. The samples were counted immediately, after 24, and after 48 hours.

The recoveries for ^{32}P were nearly 100%, regardless of the counting solution, wipe device, or the time the sample was counted. Neither the paper filter circles nor the glass fiber filters dissolve in any of the counting solutions but the glass fiber does become translucent. The plastic squares dissolved at different rates in all counting solutions – the speed of dissolution being slower in Ultima Gold XR and Opti-Fluor. In all cases, the squares appeared as either miscible or immiscible droplets on the top of the solution or on the bottom. The swab plastic stem showed different solubility patterns as well. In all cases, when the samples were shaken, they became cloudy. However, the count rates remained +/- 3% of the last count rate upon recounting. Kobayashi also suggests the use of the spectral analysis capability of the TriCarb scintillation counters as an aid to identifying various combinations of ^{3}H, ^{14}C, and ^{32}P by displaying the sample spectra in the log mode.

Similar counting efficiency studies were performed at the Battelle Pantex plant with cotton swabs, paper filters, and styrofoam squares using Ultima Gold cocktail. The results show that the best recovery of activity resulted from the addition of water or water/alcohol to the sample before the cocktail was added. As in the study done by Kobayashi, a known amount of activity (^{3}H–glucose for this study) was pipetted and dried directly on the swipe material. Except for the Styrofoam squares which dissolved, counting the swipe device by adding only cocktail resulted in lower recovery of activity than the recovery obtained when water or water/alcohol was added to the cocktail. The higher recoveries with water or alcohol/water are due to the fact that water-accepting cocktails such as Ultima Gold require the addition of a small amount of water for proper performance with water-soluble samples. The results, shown in Figure 4, were obtained within an hour of preparing the sample. The samples were not recounted at a later time.

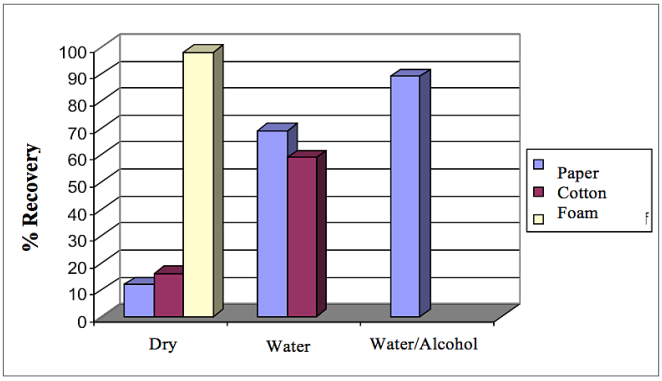

Figure 4. % Recovery of ^{3}H-surface activity. Data from Batelle Pantex

Swipe Tests for α–emitter: Counting Efficiency

Studies conducted at the Scottish Universities Research and Reactor Center (SURRC) have been performed with α–activity[6]. In this series of experiments, a grid consisting of approximately five * 5 cm squares was marked on a piece of nonporous laboratory surface material. A known amount of ^{238}Pu was pipetted onto the middle of each square and allowed to air dry for several hours. The areas were swiped with both 2 cm Whatman #1 filter paper and 2.5 cm GF/A paper (Whatman International, Maidstone, England). Triplicate swipes were taken dry, pre-wetted with 50 µl of water, or ethanol/water (50:50), and 0.1 M nitric acid. All of the samples were counted in 20 ml glass vials on a TriCarb 2550TR/AB LSC. The model 2550TR/AB is comparable with the B3110TR including the ultra low level count mode and the α–/β–discrimination option. For details about α–/β–discrimination please also read application note 17[9], "Basics of α/β–discrimination for Liquid scintillation counting". The results of this experiment are summarized in Figure 5. In all cases, pre-wetting the swipe helped to remove the activity from the surface regardless of the swiping device, with 0.1 M nitric acid giving the best results.

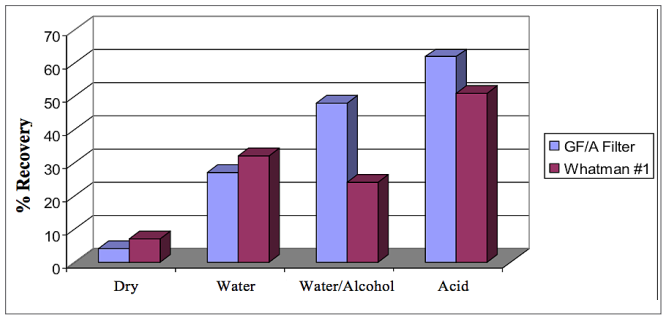

Figure 5. % Recovery of ^{238}Pu. Data from SURRC.

In another series of experiments, a known amount of activity was pipetted directly onto either Whatman #1 or GF/A paper and dried under a heat lamp. The paper was placed in the counting vial with the addition of cocktail only or cocktail plus 0.5 ml of water, ethanol/water (50:50), or 0.1 M nitric acid. Each condition was assayed in duplicate for both swipe materials.

Surprisingly, recovery of activity was nearly 100% for both types of material. However, the quality of the α–spectra differed significantly depending on the paper used and, to some extent, the agent used in addition to cocktail. The spectra obtained with the Whatman # 1 paper were the poorest regardless of the addition of water, alcohol/water, or acid. The spectral distortions observed were probably due to self-absorption of α–energy. The peaks were broad and asymmetrical, and the pulse heights were shifted to lower energy. The most symmetrical peaks observed were obtained with just the GF/A paper and cocktail was the best of all conditions tested.

Undistorted α–spectra have also been reported by Takiue et al[4]. In this study with water-soluble paper, spectra observed from ^{241}Am swipe samples show pulse height distributions similar to those obtained by homogeneous samples.

Gross α/β–Counting of Swipes

An α–reference source was prepared by pipetting approximately 1500 CPM of ^{238}Pu activity onto GF/A paper that had been soiled with dirt. Similarly, a β–source was prepared by pipetting approximately 1200 CPM of $^{90}Sr/^{90}Y$ activity onto another soiled GF/A paper disk. Three additional filters were prepared with mixed ^{238}Pu (1500 CPM) and $^{90}Sr/^{90}Y$ (1200 CPM) activity. The reference sources were counted in the α/β–discrimination mode on a TriCarb 2550TR/AB LSC (similar to model B3110 with A/B option). In this mode, the instrument discriminates light pulses produced by α–decay from those produced by β–decay on the basis of pulse decay

time and stores the α– and β–events in separate multi-channel analyzers. This pulse decay analysis (PDA) feature is used to store a percent spill curve of α– or β–event misclassification as a function of various time discriminator settings. An optimum time discriminator setting is automatically determined by the instrument at which the spill (misclassification) is minimized for both α– and β–events. Once the percent spill curve (Figure 6) is stored, it can be used as the reference curve to determine the α– and β– components of the mixed samples. In this particular case, approximately a 7.5% spill (misclassification) was calculated at the instrument determined optimum discriminator setting. Much lower misclassification is generally obtained for samples that are homogeneous. The swipe samples used in this study were prepared in a moderately soiled condition and no attempt was made to minimize any self-absorption problems.

For the mixed samples, the average error in recovery of α– and β–activity was 7%. All samples were counted for 20 minutes each. The mixed samples were counted in a 0–1000 KeV window. Although these results are not representative of all swipe assays for gross α/β–measurements, the ability to use LSC to screen swipe samples for gross α/β–activity is evident.

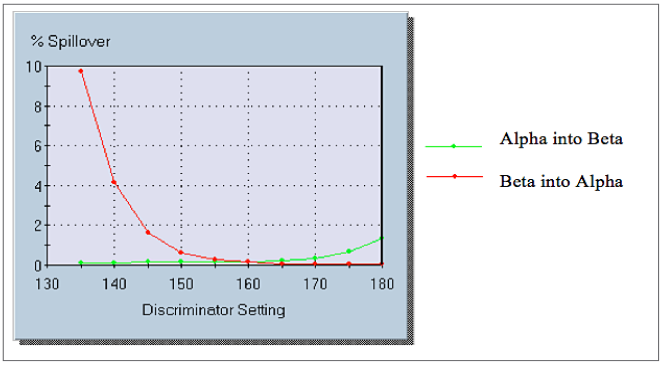

Figure 6. % Misclassification curve for ^{241}Am and ^{36}Cl in homogeneous solution.

Conclusion

Swipe assays are routinely performed to monitor surface contamination of low energy b-emitters. The U. S. Nuclear Regulatory Commission recommends taking dry swipe samples over a 100 cm^2 area. However, studies indicate that pre-wetting the swipe with water, a 50:50 alcohol/water mixture, or other agent will improve the collection efficiency. Swipe testing for α–contamination is also improved by pre-wetting the swipe material. However, pre-wetting with dilute acid will probably provide the best collection efficiencies. The only drawback to pre-wetting the swipe is that it may help spread the contamination. However, the purpose of the test is to determine the presence and severity of the contamination. Pre-wetting the swipe optimizes the collection efficiency and enhances the ability to detect the presence of any radioactivity exceeding the NRC action level of 20,000 DPM/ 100 cm^2 for restricted areas.

Contamination of a surface can be due to either water-soluble or organic soluble material. Therefore, it is essential that the counting solution solubilise either type of radioactive contaminant. It is advisable to add a small amount of water to commercially available cocktails designed to accept water-soluble samples. If the contaminant is water-soluble, the water solubilises it from the surface of the solid support and ensures good contact with the scintillation cocktail. Since a scintillation cocktail is a mixture of organic solvents, there is a good chance that an organic contaminant will also be soluble in the counting solution.

Glass fibre filters are good swipe materials for both a- and b-contamination. They have high collection efficiencies, good recovery of activity, and become transparent in liquid scintillation cocktail. However, paper filters or cotton swabs may also be used. Styrofoam plastic squares provide the lowest overall recovery of activity. Table 3 ranks, in order of performance, the overall efficiency of the various swipe media and wetting agents discussed above.

An interesting alternative to common swipe materials is the water-soluble paper reported by Takiue et al. This paper will help eliminate self-absorption of sample that is trapped on the surface of insoluble swipes, and should also have application for gross α/β–counting as well.

Also Filtercount, a cocktail especially formulated to allow dissolution of some filer types, can be used to measure filter material in homogeneous solution. As many wipe tests are done with solid papers or filters which may result in heterogeneous samples, we recommend reading application note 2 about "Filter and Membrane LS Counting"[7].

Swipes can be assayed for gross α/β–activity by liquid scintillation analyzers employing pulse decay analysis. Since it is difficult to predict the quality of sample and the type of swipe material used, a pilot experiment should be conducted with controls to insure good recovery of activity.

Table 3. Preference ranking for conventional swipe materials.

	Swipe Material	Wetting Agent
β–Contamination	1. Glass Fiber Filter 2. Cotton Swab 3. Paper Filter 4. Styrofoam	1. Water; Water/ Alcohol or Detergent 2. Dry
α–Contamination	1. Glass Fiber Filter 2. Paper Filter	1. Dilute Acid 2. Water; Water/Alcohol 3. Dry

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The application note information enclosed is used to illustrate the technique and may not represent the latest instrument, reagents and cocktails. Customers should validate the technique in their laboratory.

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