SECTION 13

WIPE TEST ANALYSIS

13.1 ALPHA BETA AND GAMMA WIPE TEST ANALYSIS

13.1.1 Scope and Applications.

13.1.1.1 Surface suspected of alpha, beta, or gamma contamination are wiped using either dry or moistened NUCON smears. The smears are then placed on the waxed surface of the NUCON smear folder and marked for identification. Wipe tests for alpha and beta activity are counted using a gas-flow proportional counter, while wipe tests for gamma contamination are counted using a gamma spectrometer.

13.1.2 Apparatus.

(A) Gas-flow proportional counter
(B) Gamma Spectrometer
(C) NUCON Smears and folders
(D) 2-inch stainless steel or aluminum planchet

13.1.3 Reagents. None

13.1.4 Calibrations.

13.1.4.1 For Beta/Alpha Wipe Tests: The sample counting efficiency is obtained by counting plated machine standards of know activity with each set of samples.

$$\text{Counting Efficiency (\%)} = \frac{\text{CPM}}{\text{DPM}} \times 100$$

CPM = Net counts per minute of the sample.
DPM = Disintegrations per minute of known activity of the sample

13.1.1.5 Procedures.

13.1.5.1 Wipe tests to be counted for alpha and/or beta activity are removed from the NUCON smear folder and placed on a prescreened (background) 2-inch planchet. A record of the planchet number is retained to provide later identification. The planchet is then placed in the gas-flow proportional counter and counted.

13.1.5.2 Wipe tests to be counted for gamma activity may be left in the NUCON folder. Individual wipe tests are then placed directly on the face of a Ge(Li) or High Purity Germanium detector for counting.

13.1.6 Interferences. None

13.1.7 Calculations.

13.1.7.1 Determine the LLD for the samples counted. Samples with values below this level are reported as less than the LLD.

$$\mathrm{LLD} = \frac{4.65\sqrt{\mathrm{CPM}_b/\ T}}{\mathrm{CE}} = \mathrm{dpm/wipe}$$

13.1.7.2 Sample results above the LLD are calculated using the following equations and reported in dpm per wipe test sample with a 2 sigma (2σ) counting error.

$$\mathrm{Activity} = \frac{\mathrm{CPM}_{\mathrm{Net}}}{\mathrm{CE}} \pm \frac{1.96\sqrt{(\mathrm{CPM}_{s+b}/T) + (\mathrm{CPM}_b/T)}}{\mathrm{CE}} = \mathrm{dpm/wipe} \pm 2\sigma$$

$\mathrm{CPM}_{\mathrm{Net}}$ = $\mathrm{CPM}_{s+b} - \mathrm{CPM}_b$

CPM_{s+b} = Counts per minute of sample plus background

CPM_b = Counts per minute of average background

T = Counting interval in minutes

CE = Counting Efficiency

2σ = 2 Standard Deviations

13.1.7.3 When activity is greater than the LLD, but the 2σ counting error is greater than the activity, report the results as the activity ± 2σ. (i.e. LLD = 1.0 and Activity = 1.3 ± 1.4(2σ), the reported results would be, 1.3 ± 1.4).

13.1.8 Sample Handling and Preservation.

13.1.8.1 A representative sample is collected using a dry or moistened NUCON smears, and a separate wipe must be used for each surface area to be wiped. The area wiped should be approximately 100 cm^2 or a 4"* 4" area.

13.1.8.2 Each wipe should be placed in a separate NUCON folder or envelope, and positively identified with a wipe number and the following information will be available:

A) Date and time wipe taken.

B) Technician taking wipe.

C) Isotope of interest, if know.

D) Name and telephone number of individual responsible for conducting tests.

13.1.8.3 Avoid touching the wipe folder(s) inside and envelope (or small zip-lock bag) seal, and mark the envelope "Laboratory Samples - Do not open."

13.2 ANALYSIS OF BETA EMITTERS

13.2.1 Scope and Application

13.2.1.1 This method covers surfaces, which are wiped using a membrane filter, suspected of low-energy beta contamination (H-3, Ni-63, C-14).

13.2.1.2 The action limit for wipe test samples of low-energy beta emitters is 1000 dpm according to Regulatory Guide 1.86. The detection limits for Ni-63, Tritium, and C-14 are approximately 3.0 x 10-6 µCi per wipe, and 3.0 x 10-4 µCi per wipe and 2.0 x 10-6 µCi per wipe respectively. If a lower detection limit is required, the sample may be counted for a longer period of time.

13.2.2 Summary of Methods

13.2.2.1 The filter is moistened with deionized water, wiped over the suspected surface, placed directly into either a prepared liquid scintillation cocktail solution or in the case of tritium wipe test, into a glass liquid scintillation vial containing deionized water. Also, in the case of Ni-63 and C-14, the filter may be placed into an empty liquid scintillation vial for shipment. Samples are prepared for analysis by adding liquid scintillation cocktail directly to the vials containing just a filter, or by taking a portion of the water from the sample shipped in distilled water, and placing it into liquid scintillation cocktail. The vials are placed into a liquid scintillation counter and dark adapted before counting.

13.2.2.2 The cocktail, when mixed with the radioactive sample, is excited by beta particles, and emits light pulses by a molecular de-excitation process. The number of pulses per unit time is proportional to the quantity of activity present. Multiple solutes are used in the cocktail to provide the best combination of wavelength and pulse height for this application. The pulses are detected by two photomultiplier tubes connected in coincidence, then converted to electrical signals. Pulses, not in coincidence, are rejected as noise. The amplified pulses are recorded and the count rate is measured. The efficiency is obtained from a quench curve, and the results are reported in μCi per wipe ± 2σ due to counting error.

13.2.3 Sample Handling and Preservation

13.2.3.1 Tritium samples, shipped in water, must have 10 ml of deionized water in the vial. This will allow for a simple aliquot correction factor to be applied.

13.2.4 Interferences

13.2.4.1 Sample type and environmental conditions can lead to very high count rates, which are not the result of radioactive decay. The high count rate can be produced by chemical reactions (chemiluminescence) and/or light (photoluminescence). Chemiluminescence and photoluminescence will cause single photons to be emitted, and they are usually rejected by the coincidence circuitry. However, if a large number of single photons are emitted simultaneously, a coincidence pulse can occur, and be recorded as a true count.

13.2.4.2 Photoluminescence and chemiluminescence have low energies, which decay over a period of time. Therefore, samples are allowed to dark adapt before counting.

13.2.4.3 The sample and the cocktail must be in a single-phase (either a clear solution or a gel). If it appears to be milky or chalky, the solution will settle into two distinct layers with time. This is a two-phase region, and it is not stable or quench correctable. Therefore, the results cannot be converted into dpm.

13.2.4.4 The filter must become transparent or dissolve after the liquid scintillation cocktail is added. If the filter does not become transparent or dissolve, heterogeneous counting will occur; this resembles a two-phase region, which is not stable or quench correctable. Also, the filter could absorb some of the beta particles, so that the particles would not produce a measurable excitation of solvent. This is a form of quenching called beta absorption, and it will result in a decrease of potential counts without any direct method to determine the degree of quench.

13.2.5 Apparatus

(A) Liquid scintillation counter.

(B) Counting vials, 25 ml, glass with screw top.

(C) Membrane filters (Metricel GN-6 or equivalent).

(D) 2.0 ml Class "A" volumetric pipet (used when wipe tests are placed in deionized water).

13.2.6 Reagents. Packard's Ultima Gold Pre-mixed Liquid Scintillation Cocktail (or equivalent). Material Safety Data Sheet is included in Appendix D.

13.2.7 Calibration

13.2.7.1 Preparation of a quench curve

(A) A quench curve enables the LS counter to convert cpm data to dpm data. The quench curve is simply a relationship between the counting efficiency of an isotope in a sample and a quench parameter (H-number or External Standard Channel Ratio (ESCR).

(B) A quench curve is derived by using data from a series of standards which are either prepared by the analyst or purchased. Each of these standards contains:

(i) The same known activity (dpm) of either Tritium, Carbon-14 or Nickel-63.

(ii) The same volume of cocktail.

(iii) Varying amounts of quenching agent, water (0.0, 0.5, 1.0, 1.5, 2.0, 2.2 ml) for standards prepared by the analyst or carbon tetrachloride for purchased standards.

(C) For the prepared standards count each vial before adding the quenching agent (water). Each vial must have the same cpm within acceptable error limits. Those vials outside the limits should be discarded.

(D) The counting error for each standard should be <1% (2σ). The counting efficiency (CE) for each standard is calculated as follows:

$$CE = \frac{CPM_{s+b} - CPM_{b}}{DPM}$$

Where: CPM_{s+b} = Count rate of standard + background
CPM_{b} = Count rate of background
DPM = Activity added

(E) The counting efficiency is plotted on the Y-axis and the quench parameter is plotted on the X-axis. The data is input to a computer statistical program. A least squares analysis and curve fit are run. The equation with the best fit is then used to determine the efficiency.

13.2.7.2 Use of the quench curve

(A) The sample is counted, and the quench parameter is measured. Then the counting efficiency is determined from the quench curve.

(B) The activity of the sample, the lower limit of detection, and the 2 sigma standard deviation are then calculated. These calculations are described in detail in subsection 13.2.9.

13.2.7.3 Commercial Quench Set

(A) The commercial quench set is good for chemical quench only (colored samples cannot be analyzed).

(B) The quench parameter ESCR is volume dependent; therefore, only analyst prepared quench curves shall be used. The sample contains 15 ml of solution plus 2 ml of sample giving a total of 17 ml but the commercial set contains 15 ml of solution only.

13.2.7.4 Internal Standardization

(A) For quench parameters outside the range of the calibration standards, the quench curve cannot be used; the internal standardization technique is used, and is described below.

(B) The sample is first counted. Then a known amount of either Tritium, Carbon-14 or Nickel-63 is added to the sample and it is recounted.

(C) The counting efficiency is calculated as follows:

$$CE = \frac{(CPM_{standard} + SAMPLE) - CPM_{s+b}}{DPM_{added}}$$

(D) The activity is then calculated as stated in subsection 13.2.7.1.

13.2.8 Procedure

13.2.8.1 For H-3 Wipe Test:

(A) For wipe test placed directly into 15 ml of liquid scintillation cocktail, place vial(s) directly into counter. Allow the sample to dark adapt, and then count for 3 minutes.

(B) For wipe test placed into 10 ml of deionized water, pipet (use a 2.0 ml class "A" volumetric) a 2.0 ml aliquot of the water, into 15 ml of liquid scintillation cocktail. Place vial(s) into counter, dark adapt, and count for 3 minutes.

13.2.8.2 For Ni-63 Wipe Test:

(A) Place wipe directly into 15 ml of liquid scintillation cocktail, (filter must become transparent before counting), place vials(s) into counter, dark adapt, and then count for 3 minutes.

(B) Use the two prepared Ni-63 standards to calculate chemical recovery (see "calculations"), and to assure the counter is functioning properly.

13.2.8.3 For C-14 Wipe Test: Place wipe directly into 15 ml of liquid scintillation cocktail (filter must become transparent before counting), place vial(s) into counter, let dark adapt, and count for 3 minutes.

13.2.9 Calculations

13.2.9.1 Recovery: For C-14 and H-3 wipe samples, the recovery is 100%. For Ni-63 wipe samples, the samples recovery is obtained by counting two prepared standards, and comparing the amount of activity added to the amount determined by the counter.

$$\%CR = \frac{(CPM_{s+b} - CPM_b)/CE}{DPM_{added}}$$

$\%CR$ = percent chemical recovery
DPM_{added} = Disintegrations per minute of standard added to cocktail
CPM_{s+b} = Counts per minute of sample plus background
CPM_b = Counts per minute of average background
CE = Counting efficiency factor from quench curve or internal standardization technique

13.2.9.2 Lower Limit of Detection:

(A) The LLD is calculated as follows:

$$LLD\ (\mu Ci/wipe) = \frac{4.65\sqrt{CPM_b/T}}{(CE)(CR)(A)(2.22 \times 10_6)}$$

(B) Sample with values below this level are reported as less than the LLD.

13.2.9.3 Sample results above the LLD are calculated using the following equation, and reported in units of μCi per wipe test sample $\pm 2\sigma$ counting error.

$$Activity\ (\mu Ci/wipe) = \frac{CPM_{s+b} - CPM_b}{(CE)\ (CR)\ (A)\ (2.22 \times 10^6)}$$

$$\underset{(\mu Ci/wipe)}{\text{Standard Deviation}} = \frac{1.96\sqrt{(CPM_{s+b} + CPM_b)/T}}{(CE)(CR)(A)(2.22 \times 10^6)}$$

Where: CPM_{s+b}, CPM_b, and CE are as defined in subsection 13.2.9.1

And: CR = chemical recovery (calculated for Ni-63 only)
A = aliquot correction factor (usually 0.2, when a 2.0 ml aliquot is used from a 10. ml sample)
T = counting time in minutes (sample count time must be equal to background count time!)
μCi/wipe = microcurie per wipe

13.2.9.4 When the activity (1.3) is greater than the LLD (1.0), but the 2σ counting error (1.4) is greater than the activity, the results are reported as activity ± 2σ (1.3 ± 1.4).