

JEDEC STANDARD

Alpha Radiation Measurement in Electronic Materials

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ALPHA RADIATION MEASUREMENT IN ELECTRONIC MATERIALS

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Introduction

Soft error upsets in semiconductor devices are caused by energetic particle interactions with the sensitive nodes in the device. One source of these energetic particles is radioisotope impurities in the materials that comprise the device. Alpha particles are of primary concern, and materials that have low alpha activity have been selected for critical applications to mitigate this effect. Measurement of the alpha flux is important to establish both the usability of these materials and the reliability of the semiconductor devices fabricated from them.

The measurement of alpha flux below $10 \alpha \cdot \text{hr}^{-1} \cdot \text{cm}^{-2}$ is complicated by the fact that the sample alpha flux is usually less than or equal to the background alpha flux in the detector. Achieving a reasonable degree of precision requires measurements lasting for many hours or days. The low signal to background ratio also makes measurement results vulnerable to variations in techniques and methods. Elimination of or compensation for these sources of measurement variation allows for scientifically and statistically valid results that are reproducible between different laboratories.

ALPHA RADIATION MEASUREMENT IN ELECTRONIC MATERIALS

(From JEDEC Board Ballot JCB-11-21, formulated under the cognizance of the JC-13.4 Subcommittee on Radiation Hardness and Assurance and the JC-14.1 Subcommittee for Reliability Test Methods for Packaged Devices.)

1 Scope

This standard applies generally to gas proportional instruments and the use thereof in measuring materials with an alpha emissivity of less than $10 \alpha \cdot \text{hr}^{-1} \cdot \text{cm}^{-2}$. The primary focus will be on materials used in semiconductor fabrication.

The purpose of this document is to specify the recommended method for measuring alpha emissivity in materials utilized in the manufacturing of semiconductors. The method specifically applies to gas proportional instruments and designates recommended instrument settings. In addition, the method discusses operation of ionization counters. The document also recommends methods for determining sample size and for evaluating instrument background accurately. Treatment of data is also outlined, including identification and elimination of systematic errors. The calculation of results and detection limits is detailed with examples in the annexes. A standard format for reporting results is specified.

2 Terms and Definitions

accuracy: A measure of how close the measured result is to the true value.

bias voltage: Potential applied between the anode and cathode in a gas proportional counter.

detector background: Signal measured by the detector in the absence of a sample.

discriminator: Signal rejection mechanism which eliminates low and high energy events.

efficiency: The ratio between the number of alpha particles detected and the actual number of events occurring. This same value for the detector efficiency is used when measuring the detector background.

emissivity: The rate of emission of alpha radiation measured in counts per unit area per unit time.

2 Terms and Definitions (cont'd)

gas proportional counter: Instrument which detects radiation by measuring ionization of counting gas between two electrodes. The anode is usually comprised of a fine wire or grid of wires so that the local electric field near the wires is large enough to cause gas multiplication and thus a large signal. A proportional counter has an output pulse proportional to the number of primary ions produced in the counting chamber by the sample radiation.

ionization counter: Instrument which detects radiation by measuring ionization of counting gas between two electrodes at lower voltages below what is necessary for gas multiplication.

limit of detection: The minimum alpha flux emitted from a sample that is statistically significant above the background at a defined confidence level.

poisson distribution: Statistical distribution that predicts discrete variable behavior, including alpha particle counting.

precision: The degree of mutual agreement among data that have been obtained in the same way.

quenching: The technique used to control ionization in a gas proportional counter by addition of an electropositive gas which absorbs secondary electrons.

systematic error: Spurious events detected by a counting system which do not originate in the sample and are not random.

3 Instrument Operation Parameters

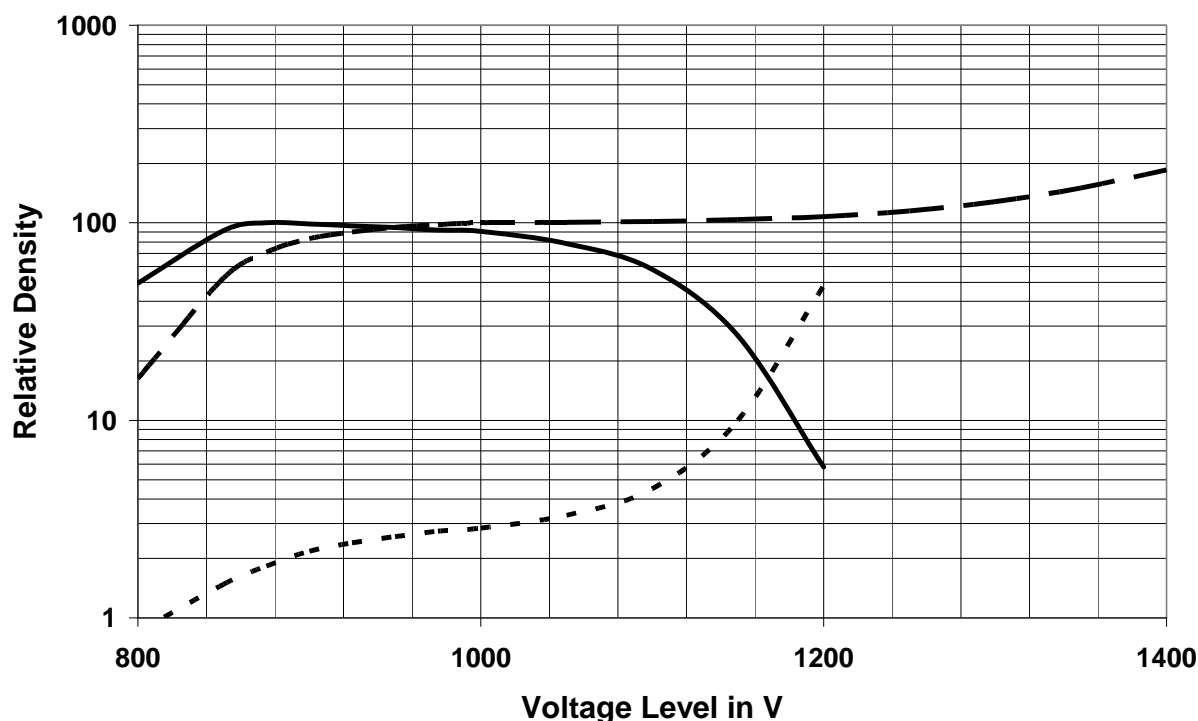
For a good reference on proportional counter operation, see Knoll¹. A discussion on a modern ionization counter, with pulse shape discrimination, and low background is given by Warburton², and Gordon³. In the following sections, the discussion generally refers to proportional counter operation.

3.1 Bias Voltage

A standard setting for the applied bias voltage is not practicable given the differences in individual detector design and construction. What is desired is that the electric field between anode and cathode in a gas proportional counter be large enough to ensure that multiplication occurs. Therefore, the bias voltage might vary instrument to instrument depending on the anode-cathode distance, the gas type, wire diameter, and gas pressure within the instrument.

3.2 Bias Point Selection

A recommended method for selecting the bias voltage for proportional counters is outlined by Schindlbeck⁴. The absolute efficiency and background count rate of the instrument is measured as a function of increasing bias potential. These two values and their ratio (relative efficiency) are plotted in Figure 1. The optimum bias voltage occurs at the maximum of the ratio between the absolute efficiency and the background count rate. In this region the relative efficiency represents the best balance between increasing signal and decreasing noise. In this example the optimum voltage is 900 volts.



Dashed line = counting efficiency dotted line = background counts
solid line = relative efficiency (= counting efficiency / background counts)

Figure 1 — Example bias point selection graph.⁴

Another method is to measure the pulse height from the amplifier, using an oscilloscope or a multichannel analyzer, as a function of the bias voltage. The voltage should be large enough to ensure that the pulse height varies exponentially with applied voltage.

3.3 Discriminator

The discriminator defines the range of alpha particle energies that are counted, and affects the measured count rate and the calculated emission rate from the sample. At energies less than 1.2 MeV background noise from beta radiation becomes significant, so the recommended discriminator threshold is 1.2 MeV. For vendors providing raw materials this alpha energy range (higher than 1.2 MeV) is recommended as it makes no assumptions about the final use of the material in a product. If specific instrument limitations result in significant noise above 1.2 MeV, the lower threshold will be adjusted accordingly, and the revised energy range stated in the report.

The lower level discriminator could be set by measuring the count rate and energy spectrum of a thick source (NIST traceable) using a standard solid state detector (i.e., silicon) in a 2π geometry configuration. Once the energy spectrum and count rate over the applicable energy range is determined, the same source is placed in the proportional or ionization counter and the lower discriminator is adjusted to give an equivalent count rate response.

For many semiconductor products, alpha emission from the packaging materials (external to the silicon chip) is one component of soft error upsets. Alpha particles emitted from the packaging materials must traverse many metal and dielectric layers to reach the active silicon circuitry, and lose energy in proportion to the material density and distance traveled. Semiconductor device manufacturers may desire to account for the fact that lower energy alpha particles (above the discriminator energy) will be harmlessly absorbed in the chip layers before reaching the device silicon. In such cases the discriminator setting can be set higher so that only those alpha particles able to cause a soft error in the actual chip are counted. Figure 2 shows the alpha particle range as a function of energy through copper and SiO₂ and the threshold at which a soft error is initiated.

The energy to be used should be defined by a conservative estimate of the minimum energy that could traverse the chip layers without being absorbed. For a conservative estimate, normal incidence is used (since at higher angles the effective distance traveled is greater) and the alpha reaching the silicon with 0 MeV remaining is the threshold (when in actuality, an alpha particle needs some energy to create a soft error). To keep the estimate conservative and to account for the fact that minimum metal coverage due to interconnect is usually 20% we can calculate an effective thickness of metal as the product of the actual metal thickness in the chip and the coverage fraction. For the dielectric layers one can use the actual thickness of the layers. This is not very accurate but does ensure that the minimum energy defined is conservative with no possibility of underestimating the alpha particle flux reaching the device surface. More accurate methods based on ray tracing through actual layers with interconnect layout considerations can be done with simulations to get a better estimation of the minimum discriminator setting. Irrespective of the actual technique used the minimum discriminator energy must be recorded in the final report of the alpha emission for the sample.

3.3 Discriminator (cont'd)

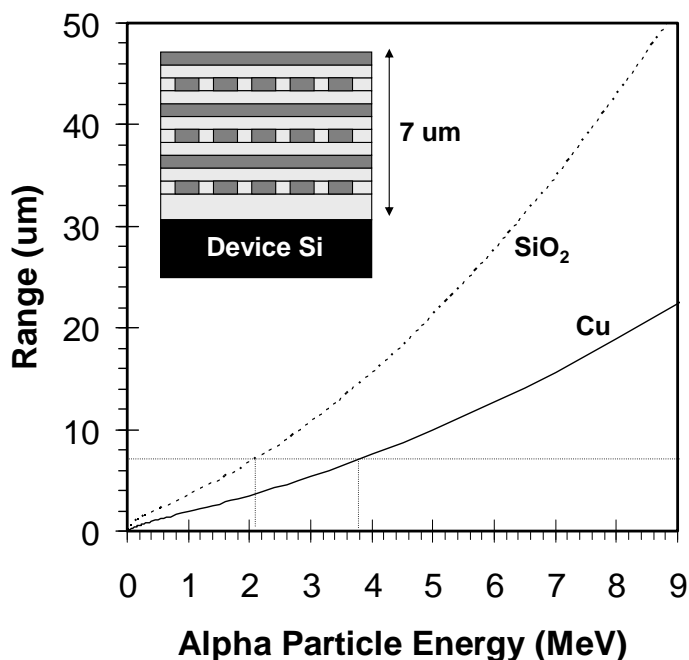


Figure 2 — The range of alpha particles in semiconductor materials with sample chip cross-section demonstrating the range of minimum discriminator energies for a 7um stack that is 100% SiO₂ or 100% Cu.⁵

3.4 Gas Management and Maintenance

3.4.1 Gas Type

The gas type used shall be reported. Typical proportional counting gases include P-10 and P-5. There is a negligible difference in results between these two gases. If other counting gases (Ar-CO₂, Isobutane) are used, differences in quenching must be compensated for when comparing settings from instruments utilizing P-10 or P-5. In ionization counters, counting gases can include Ar or N₂ without the need for a quench gas.

3.4.2 Gas Purity

Standard high purity counting gas is recommended over ultra high purity gas because the difference in performance of gas counters is negligible for these applications. It is recommended that counting gas quality be verified periodically to eliminate the possibility of using radon contaminated counting gas. A recommendation to quantify gas quality consistency is to measure background or a check sample after each cylinder change, and examine the before and after results for shifts in count rate. A gas cylinder should only be used after 20 days have passed from filling to allow any radon introduced to decay.

3.4 Gas Management and Maintenance (cont'd)

3.4.3 Optimal Gas Flow

For unsealed proportional counters, the gas flow must be large enough to prevent any intrusion of ambient radon or water vapor. Radon will lead to elevated count rate, and any vaporized electronegative compounds (water, alcohol) will influence gas multiplication or ionization. The gas flow rate will be specific to each individual instrument, and should be the volume flow rate necessary to give optimum efficiency and background. A typical value may be 200 to 300 $\text{ml}\cdot\text{min}^{-1}$. For sealed gas flow chambers where ambient gas intrusion is not an issue, the recommended minimum flow is 100 $\text{cc}\cdot\text{min}^{-1}$. It is also recommended that when not in operation the chamber is purged with high purity counting gas or N_2 to minimize contamination infiltration.

The gas volume in ionization counters is typically much larger than that for proportional counters. The signal amplitude in ionization counters is much smaller than proportional counters, and discrimination between alpha particles emanating from the anode or sample rely on the distance between the anode and cathode being larger than the range of alpha particles in the counter gas.

After a sample change, the ionization counter may need to be purged- a period of higher than normal gas flow rate to displace the oxygen and/or radon that was introduced. Rise times shorter than normal may result if the counter has not been purged for sufficient time. Similarly, the ionization counter might require a larger gas flow rate than the proportional counter.

3.4.4 Gas Tubing Specification

All plastic tubing is to some degree permeable to radon, so metal tubing should be used instead. However, the metal tubing should be electrically isolated from all metal parts of the counting system to avoid antenna effects. The recommended configuration is to have a short (10 cm) length of polyethylene tubing connecting the metal tubing to the detector.

3.4.5 Active Area Determination

Large active area detectors are necessary to accurately measure the alpha flux in low alpha materials. The detector active area has a significant and direct effect on the accuracy and precision of the calculated alpha emissivity. The efficiency around the periphery of some gas proportional instruments may decrease due to electric field changes and/or the escape of alpha particles emitted away from the active volume. (See Figure 3 and Reference 4). In such an instance, any decrease in efficiency over the entire detector area may be compensated by:

- 1) Decreasing the sample area to correspond to the area over which the efficiency is not less than 95% of the calibration efficiency in the center, or
- 2) Using the average efficiency over the entire sample area, not the efficiency from the center.

3.4 Gas Management and Maintenance (cont'd)

3.4.5 Active Area Determination (cont'd)

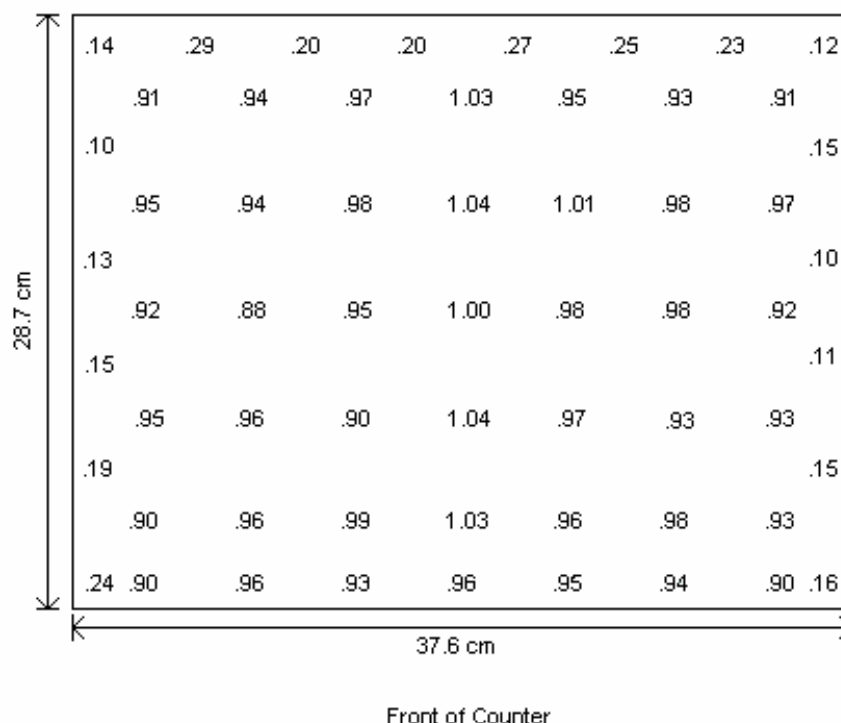


Figure 3 — Example of gas proportional counter efficiency at different positions

3.4.5.1 Method for determining efficiency as function of area

Samples of varying areas (i.e., 600, 700, 800, 900, and 1000 cm²) are made from a moderate activity material ($\sim 100 \alpha\text{-khr}^{-1}\cdot\text{cm}^{-2}$). Each sample is measured and the alpha emissivity normalized for sample area is calculated. Determine at which area normalized results differ. For standard grid proportional counters, this will typically occur in the outer 100 cm² area. If all results agree to within measurement uncertainty, the detector efficiency is consistent over the entire area. An example is found in Annex A.

Alternatively, a small area (point) Thorium or Americium source can be used to measure efficiency at a number of locations across the detector area. The efficiency over the entire area is calculated by taking an average of the individual locations across the detector (See example calculation in Annex A). Increasing the number of locations gives a better estimate of the detector efficiency. This method requires shorter measurement times, and avoids effects of poor homogeneity in the large area sheets.

3.5 Sample Distance

For instruments with window electrodes, the sample/window distance shall be 2 - 3 mm.

4 Calibration

Instrument calibration shall be performed under normal operating conditions. The appropriate standard is placed the same distance (2-3 mm) from the sensitive region in the center of the detector. The horizontal and vertical position may be reproduced by marking the appropriate position on the tray. For the purposes of this standard document, calibration is internal to each organization. There is currently no generally accepted external calibration standard suitable for this application. However, ^{232}Th or ^{241}Am have been used by a number of laboratories.

4.1 Calibration standard

Thick sources with a continuous energy range from zero to the maximum energy are recommended for standards. The calibration standard emissivity should show no significant change over time and have a sufficient count rate to yield reasonable counting statistics over the calibration run. The efficiency is calculated as the ratio of the detected count rate to the certified count rate.

Since all calibration activities described in this standard are internal to each individual organization, equivalence between different laboratories or instruments must be established before any comparison of sample results is valid. This equivalence is quantified by counting a sample at the respective laboratories or in the respective instruments and examining the results for bias. If the results are not statistically different, no bias exists and equivalence is established. If the difference in comparison results is statistically significant, this bias must be considered when comparing results from the different laboratories or instruments.

4.2 Calibration Interval

Although gas proportional counters are very stable relative to other analytical instrumentation, calibration should be performed at least annually. Calibration may be done more frequently as required by internal procedures or customer requirements. In the event of instrument maintenance or malfunction, calibration must precede return to normal utilization. The calibration date shall be recorded.

5 Background Measurement

The measurement and subtraction of background is a critical step in low alpha materials measurement, and the importance of correct background determination cannot be overemphasized. Since the background signal is in most cases comparable to or larger than the sample signal, a small error in background count rate leads to a significant error in the end result. In general, the instrument background should be the same in the presence or absence of a sample.

5.1 Trays or sample stages

In the gas proportional instruments of interest, the sample is placed on a tray or stage, which is then used to position the sample within the detector. The specification requires that the sample tray or stage be positioned in the analysis chamber during background measurement. In order to meet the requirement that the background should be independent of the presence/absence of a sample, the alpha contribution from the surface of the tray or stage should be below the detection limit. The alpha contribution of the tray must be checked periodically. Measuring background with the tray after a sample is removed will determine if the material has introduced contamination. Background data may also be used to detect upward trends or cross contamination (see Figure 4). If inconsistent, the tray surface must be cleaned to remove any contamination or must be shielded during background measurement. Materials with alpha emissions at or below the nominal detection limits include silicon wafers, polycarbonate, and clear acrylic plastic.

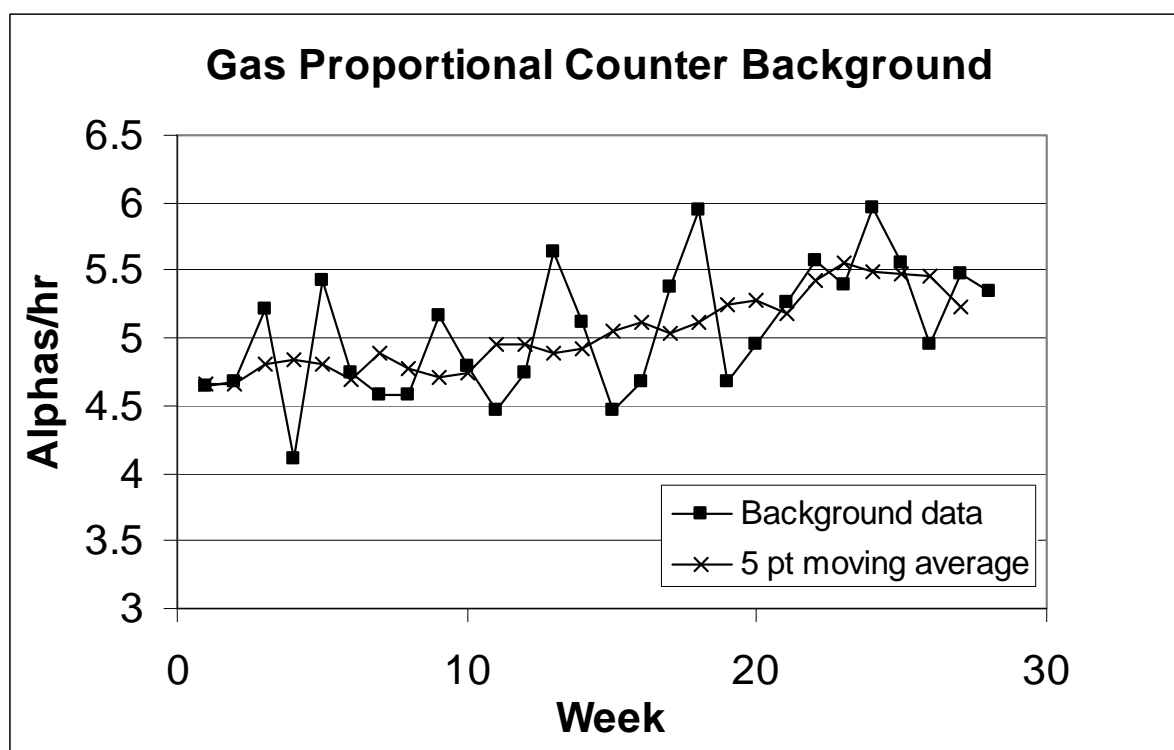


Figure 4 — Background data and 5 point moving average

5.2 Method of Determining Background

5.2.1 Measure background before each analysis

One method of determining background is to measure the chamber background for a given period of time immediately before, and after, the sample is introduced. The time interval is equal to or greater than the analysis time interval. This method assumes the background variation over the time intervals is negligible. Shorter background counting times could be used, but these would be less representative of the background over the analysis time. To verify a consistent background in this case, the background would need to be measured immediately after the assay. If the variation between the before and after background is statistically negligible, the background is proven to be consistent, and the average of the two is the best estimate of the background. This method is recommended if the background is very gradually (0.1 to 0.2 counts per hour) increasing over an extended period of time (i.e., 1 year).

5.2.2 Long term average of statistically controlled background

Another estimate of the instrument background is a moving average of a number of background measurements. In this method, the variation in the background is monitored by statistical process control by counting background for a given time (i.e., 48 hours) at a given interval (i.e., every 10 days) and plotting the results on an X-bar chart (Figure 5). The individual background measurements are normally distributed about a mean with a standard deviation. If all points are within $\pm 3\sigma$ of the mean, each result is a valid measure of the background. The average of these points is the best estimate of the background given the long term stable behavior of the instrument. A 30 point moving average is recommended. The time over which the background was measured is recorded.

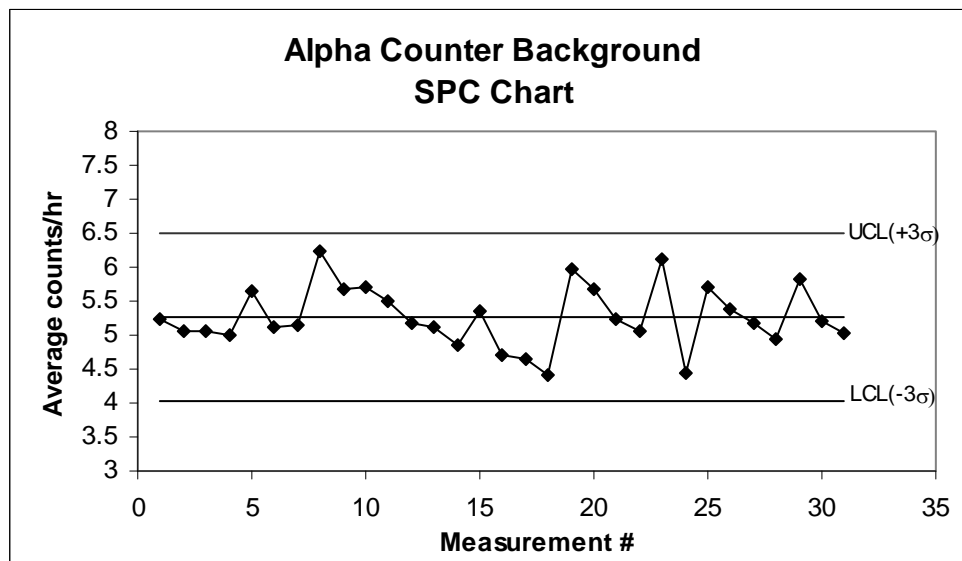


Figure 5 — X-bar chart of instrument background

5.3 Background measurement time requirement

The graphs from Figure 6 may be used to determine counting time required to achieve the desired detection limit. The 90 percent confidence (1.64σ) detection limit for samples with both 1000 cm^2 area (left) and 400 cm^2 area (right) and 80% detector efficiency are plotted versus the counting time for different background levels. The counting time should be adjusted for differences in sample area, detector efficiency, and background if the parameters differ from those on the graph.

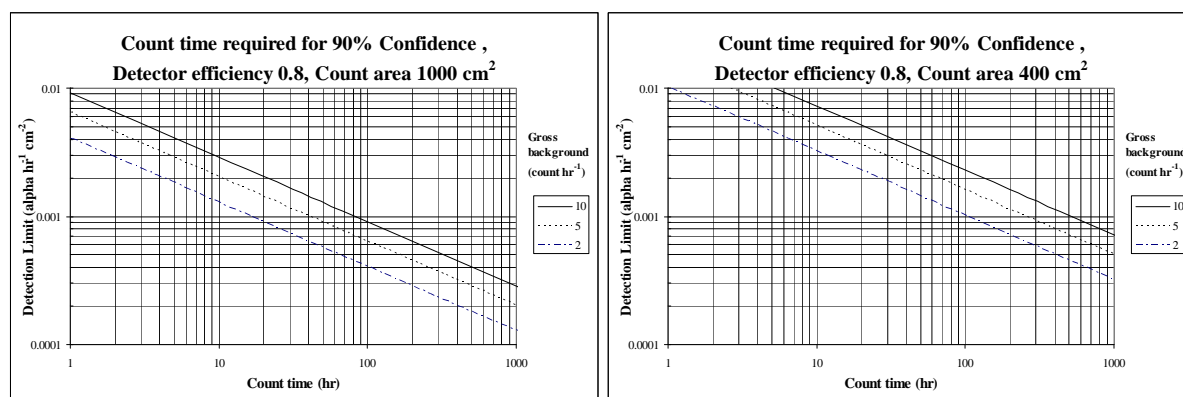


Figure 6 — Detection limit as a function of instrument background

6 Detection and treatment of systematic errors

As with all analytical instrumentation, gas proportional counters sometimes register spurious results. The identification and elimination of these false signals is necessary for accuracy and reliability. Since alpha decays are discrete events, alpha counting follows Poisson statistics. Two methods may be used to detect systematic errors. Both are based on Poisson counting statistics. For a simple, qualitative inspection of the data, the F statistic test is adequate. The recommended method is the cumulative density function which is a more thorough and conclusive technique for identifying systematic errors.

6.1 Cumulative Density Function

Since counting rates follow a Poisson statistics, the slope of the cumulative density function (CDF) depends only on the mean value. Any other slope indicates that the measured counting rates do not follow a Poisson distribution. This would most likely be caused by a systematic error influencing the measurement. Consequently the standard deviation would be wider than the case with no systematic error. The CDF of measurement results should be plotted on a grid of Poisson distributions. An example is shown in Annex B.

6.2 F Statistic or ratio of variances

A similar method is to compare the variance of the observed hourly count rate data with the expected Poisson variance. This calculated as

$$F = \frac{\sigma^2}{\bar{X}}$$

where σ is the standard deviation of the hourly count data and \bar{x} is the mean count rate. If the ratio is unity, the variability in the hourly count rate data is attributable to Poisson statistical variability, and no systematic error is present. A value appreciably greater than 1 indicates data variability in excess of Poisson statistical variation, the source of which is systematic. A value considerably less than 1 suggests that not enough variability is present, the cause of which is most likely instrumental. As a general rule of thumb, values ± 0.3 from 1 warrant further investigation.

6.3 Treatment of outliers

No data shall be rejected unless there is a specific reason for the data to be corrupted, even though it might be much larger or smaller than adjacent hourly data. An example of such a case would be if several instruments register an hourly count increase simultaneously due to a power transient. Extended analysis times like 100 hours minimize the effect of the outliers. If data are rejected, the reason must be specified in the final report.

6.4 Method for determining count rate stabilization and initial data rejection

When the chamber is opened to insert a sample, ambient gases, including radon, infiltrate into the chamber. The result is an increased count rate that decays down to normal levels, often in a matter of hours. The time required to reach the nominal count rate after chamber exposure to the atmosphere varies depending on the amount of atmospheric gas intrusion, the structure of the sample, the ambient activity, humidity, and other factors.

The hourly count rates are recorded, and the data are considered valid when the background and sample have reached nominal count rates. The hourly count rates may be considered stable when each successive value of a five point moving average does not differ statistically from the preceding value (see Figure 9 in Annex C for an example). In unsealed counters, this can take 5-10 hours in general for solid samples. For powder samples, count rate stabilization may take 24 hours or longer due to atmospheric gas inside the powder that is not readily flushed with counting gas.

Two other circumstances warrant special consideration. First, if a sample tray being used has had prolonged atmospheric exposure immediately prior to the assay, the count rate could take a significant amount of time to return to normal because of surface effects (dust, adsorbed radon). Second, if the tray has been cleaned with a solvent (i.e., 2-propanol), care must be taken to ensure the solvent has completely evaporated before insertion into the counter.

6.4 Method for determining count rate stabilization and initial data rejection (cont'd)

Some errors commonly observed are cataloged in Table 1, along with the root cause of the error and the method of correcting the error.

Table 1 — Common error sources and corrections

Observed Error	Probable Cause	Recommended Correction
Alpha Contamination	Sample left on sample tray for extended period (especially powdered samples)	Thoroughly clean tray after use with soap and water or propanol
	Pick up from materials used in cleaning and preparation	Whenever possible qualify material for use by alpha counting
	Using abrasive cleaners	Avoid abrasives when cleaning. Use only high purity solvents.
	Sample residue accumulates in the sample chamber	Periodically clean the sample chamber by swabbing the interior of the chamber with a propanol soaked cloth. Extreme care must be used when cleaning the mylar window.
High alpha count on initial readings	Static charge on mylar or sample tray attracting charged radon decay daughter particles or radon adsorbed on sample surface	Avoid buildup of static charge by grounding tray before adding sample and before placing into sample chamber. Charge may also be removed by placing a grounded conductive sheet in contact with the tray surface and then removing prior to analysis.
	Ignoring 10-hour radon daughter decay	Use 5 point moving average to determine count rate stabilization
High Background	Power supply spikes	Filter power supply
	High radon environment	Increase flush time and or rate
	Insulating materials that charge up and getter radon daughters (e.g., chip underfill)	Avoid static charge buildup by grounding sample and tray
	Counting window damage	Replace window
	Oxygen, water, or other organic in P10/P5	Replace gas source
Background corrected count rate is greater than 1 sigma below zero	Background subtraction too large	Use correct background subtraction, or measure background again and clean tray as needed.

7 Detection Limits

The detection limit is calculated as a multiple of the standard deviation

$$LOD = n\sigma = n \frac{\sqrt{\frac{G}{t_g^2} + \frac{B}{t_b^2}}}{A\varepsilon} \quad \text{Equation 1}$$

where G and B are the gross sample counts and background counts, t_b and t_g are the background and measurement counting times, A is the sample area, and ε is the detector efficiency. The confidence interval is determined by the value of n. The common definition of the detection limit is at $n = 3$, or 99.7 % confidence. The detection limit at 90 % confidence ($n = 1.64$) has also been used. The detection limit and confidence interval shall be quoted with the result, and will be determined by internal and/or customer specifications. An example of the computation of the emissivity, and error propagation for a representative data set is shown in Annex C. The dependence of detection limit on the counting time and background count rate is shown in Figure 6.

8 Secular Equilibrium Considerations

In certain cases the sample activity will increase significantly over time. In the case of lead materials, ^{210}Pb is a ^{238}U daughter that beta decays to produce ^{210}Po , which decays to ^{206}Pb by emitting a 5.4 MeV alpha particle. During processing, volatile ^{210}Po is removed, causing an initial decrease in activity. As ^{210}Po concentrations are replenished by ^{210}Pb decay, the activity will increase until the activity of the parent ^{210}Pb and daughter ^{210}Po become equal, a condition known as secular equilibrium. An increase in sample activity over time after chemical or metallurgical processing is evidence for secular equilibrium. For $^{210}\text{Pb}/^{210}\text{Po}$, alpha emissivity due to secular equilibrium reaches a maximum 27 months from the time of processing. Evidence for an increase in activity may not be conclusive in the initial weeks following processing, so the recommended elapsed time is 90 days or longer for lead containing samples. Certification of results for lead materials shall include reporting the time elapsed between processing (i.e., solder bump plating) and measurement.

9 Uniform Method of Reporting Data

Report to include:

- Sample Identification
- Counter Identification
- Counter Active Area/Sample Area
- Energy Range of Detected Alpha Particles (if known)
- Counter Efficiency
- Sample/Electrode distance if different from nominal
- Gas type/Flow Rate
- Counter Background
- Gross Alpha Flux
- Average Alpha Emissivity
- Standard Deviation of Alpha Emissivity
- Detection Limit and confidence level
- Count time
- Elapsed Time from material processing to the middle of the measurement period (for secular equilibrium materials)

10 References

1. Knoll, G. F, Radiation Detection and Measurement, 3rd ed. John Wiley & Sons (New York) 2000.
2. W.K. Warburton, Brendan Dwyer-McNally, Michael Momayezi, and John E. Wahl, "Ultra-low background alpha particle counter using pulse-shape analysis", paper #N16-80 in IEEE Nuclear Symposium Conference Record 2004, Vol.1,577-581 (16-22 Oct 2004).
3. Michael S. Gordon, David F. Heidel, Kenneth P. Rodbell, Brendan Dwyer-McNally and W. K. Warburton, "An evaluation of an ultra-low background alpha-particle detector", IEEE Transactions on Nuclear Science, vol. 56, no. 6, pp. 3381-3386, Dec. 2009.
4. Schindlbeck, G. Journal of Electronic Materials, 29(10) 2000, 1284-1289.
5. The alpha particle range in elements or compounds can be determined using SRIM- see <http://www.srim.org/>.

Annex A (normative) Active area determination

A.1 Example 1: Uniform efficiency over the entire detector area

Sample Area (cm ²)	Measured Counts/cm ²	Actual Counts/cm ²	% Efficiency
500	200	250	80
600	240	300	80
700	280	350	80
800	320	400	80
900	360	450	80
1000	400	500	80
*Material Activity = 1000 α·khr ⁻¹ ·cm ⁻² , 30 minute count time			

A.2 Example 2: Non-uniform efficiency over entire area

Sample Area (cm ²)	Measured Counts/cm ²	Actual Counts/cm ²	% Efficiency
500	200	250	80
600	240	300	80
700	280	350	80
800	300	400	75
900	320	450	71
1000	330	500	66

In Example 1, the efficiency is consistent over the entire detector area. Therefore, an efficiency of 80% may be used to calculate the result for a 1000 cm² sample.

For Example 2, the efficiency decreases for areas larger than 700 cm², so using an efficiency of 80% for a 1000 cm² sample will give an erroneous result. The correct result is obtained by using the efficiency corresponding to the sample area. In the case of a 1000 cm² sample, the correct efficiency would be 66%. If the user wishes to use 80% efficiency, then the sample area must be confined to 700 cm² or less.

A.3 Example 3: Calculating average efficiency for a large area from point source data

In this example, the 1000 cm² area in a counter is divided into 40 equal square sectors with dimensions of 5 cm by 5 cm. A point source is placed at the center of each sector and the efficiency measured. This approach assumes that the efficiency is constant over the sector area. The resulting data are shown in Figure 7.

0.79	0.79	0.84	0.88	0.88	0.83	0.85	0.75
0.80	0.75	0.90	0.87	0.81	0.85	0.84	0.78
0.79	0.77	0.85	0.83	0.86	0.85	0.87	0.85
0.86	0.80	0.85	0.87	0.83	0.83	0.87	0.78
0.78	0.80	0.87	0.87	0.89	0.81	0.82	0.82

Figure 7 — Example of point source efficiency over 1000 cm² counter area.

The efficiency for the entire 1000 cm² area is calculated by taking the average of the individual sector efficiency. For this case, the average of the forty individual sectors is 0.83.

Annex B (normative) Cumulative Distribution Function Example

Table 2 lists the hourly counting rates of a 100 hour measurement. Table 3 lists the different count rate values, the frequency with which they occurred (density), and the cumulative density. Dividing by the duration of measurement in hours gives the column “cumulative n/t”. These values are plotted on a Poisson grid, shown in Table 4.

The first column of the Poisson grid lists all the values of hourly rates ($= x$). The next 10 columns show the CDF of Poisson distributions with mean values from 1 to 10 for hourly rates $= x$ ranging from 1 to 10. The last column is the result from Table 3. The CDF is displayed by plotting these data together in the “Poisson grid”. The measurement results fit in well, indicating no obvious systematic error (Figure 7). Figure 8 shows the CDF from another measurement in the Poisson grid. These data deviate significantly from the Poisson grid, indicating that the measurement has systematic errors.⁴

Table 2 — Hourly Count Data

2	7	2	4	1	2	4	3	3	1	4	1	2	3	3	3	3	6	2	3
3	2	6	1	4	4	3	2	5	2	4	2	9	4	4	6	0	3	4	7
3	4	4	2	5	6	7	4	2	2	1	5	3	2	2	2	4	4	5	5
5	3	7	5	3	3	5	1	4	3	1	4	0	1	4	2	2	5	3	3
1	5	5	6	4	0	3	8	5	6	5	3	2	1	1	6	3	3	2	8

Table 3 — Count Rate Frequency and Cumulative Density

x	n	cumulative n	cumulative n/t
0	3	3	0.03
1	11	14	0.14
2	19	33	0.33
3	22	55	0.55
4	18	73	0.73
5	13	86	0.86
6	7	93	0.93
7	4	97	0.97
8	2	99	0.99
9	1	100	1
		t = 100	cumulative density

Table 4 — The Poisson Grid

x											Example
0	0.368	0.135	0.05	0.018	0.007	0.003	0.001				0.03
1	0.736	0.406	0.199	0.092	0.04	0.017	0.007	0.003	0.001		0.14
2	0.92	0.677	0.423	0.238	0.125	0.062	0.03	0.014	0.006	0.003	0.33
3	0.981	0.857	0.647	0.434	0.265	0.151	0.082	0.042	0.021	0.01	0.56
4	0.996	0.947	0.815	0.629	0.441	0.285	0.173	0.1	0.055	0.029	0.73
5		0.983	0.916	0.785	0.616	0.446	0.301	0.191	0.116	0.067	0.86
6		0.995	0.966	0.889	0.762	0.606	0.45	0.313	0.207	0.13	0.93
7			0.988	0.949	0.867	0.744	0.599	0.453	0.324	0.22	0.97
8			0.996	0.979	0.932	0.847	0.729	0.592	0.456	0.333	0.99
9				0.992	0.968	0.916	0.83	0.717	0.587	0.458	1
10				0.997	0.986	0.957	0.901	0.816	0.706	0.583	

Annex B (normative) Cumulative Distribution Function Example (cont'd)

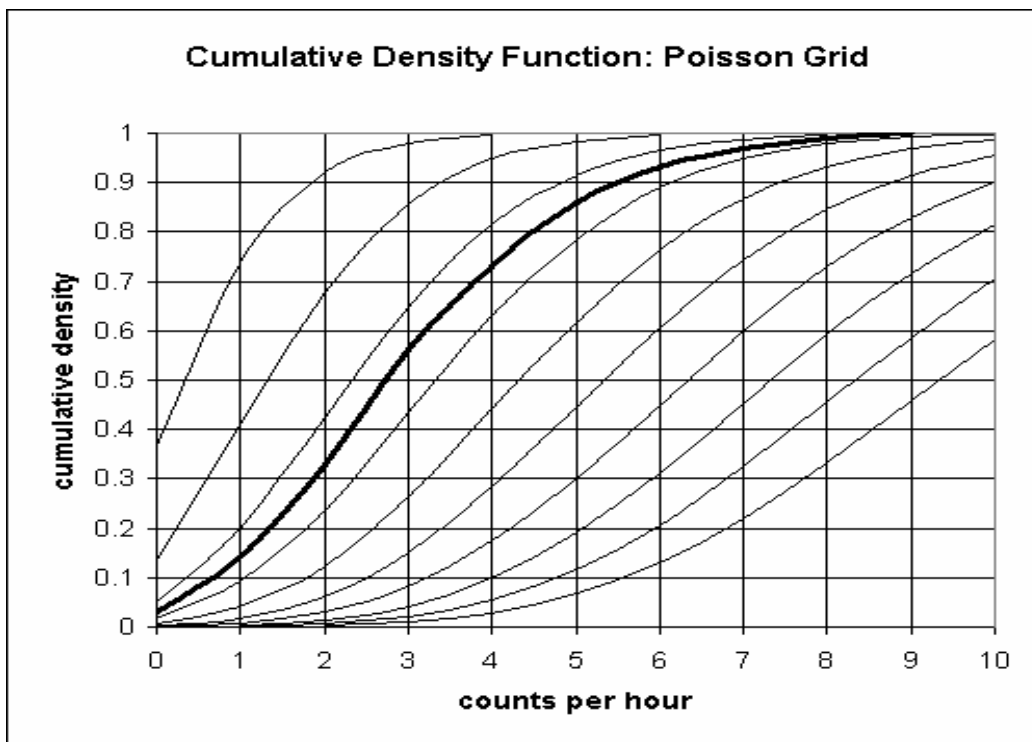


Figure 8 — CDF graph of counting data without systematic error⁴

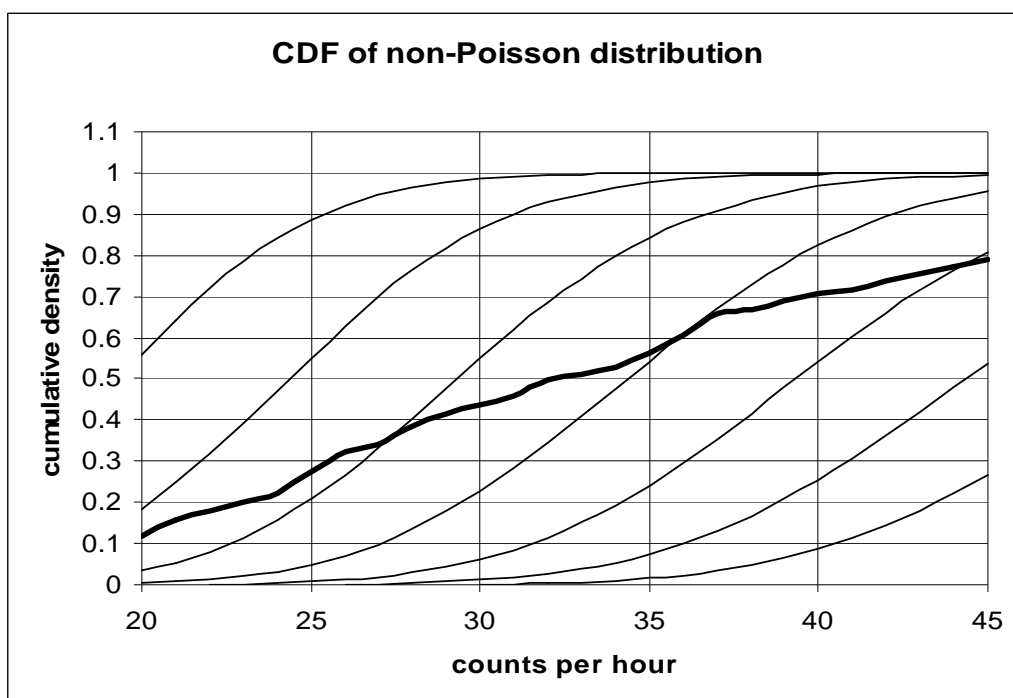


Figure 9 — CDF graph of counting data with systematic error⁴

[illegible]

Annex C (informative) Sample Calculation

The background and sample data from a typical measurement are displayed in the two tables below. The treatment of the data and the stepwise calculations used to arrive at the final result follow the tables.

Background Data

Hour	Count	Hour	Count	Hour	Count
1	225	31	4	61	7
2	68	32	1	62	4
3	31	33	4	63	2
4	15	34	5	64	3
5	5	35	4	65	2
6	4	36	5	66	7
7	5	37	5	67	2
8	4	38	3	68	7
9	1	39	6	69	5
10	7	40	6	70	4
11	4	41	6	71	5
12	5	42	5	72	5
13	8	43	5	73	1
14	6	44	4	74	1
15	3	45	4	75	0
16	5	46	3	76	1
17	9	47	4	77	6
18	8	48	5	78	3
19	3	49	3	79	3
20	3	50	7	80	2
21	6	51	1	81	2
22	4	52	2	82	1
23	3	53	3	83	3
24	3	54	0	84	7
25	5	55	5	85	3
26	5	56	6	86	2
27	6	57	4	87	1
28	2	58	3	88	2
29	3	59	6	89	8
30	3	60	7	90	4

Sample Data

Hour	Count	Hour	Count	Hour	Count
1	171	24	5	47	5
2	57	25	4	48	0
3	19	26	4	49	4
4	6	27	10	50	6
5	7	28	1	51	8
6	6	29	6	52	10
7	10	30	4	53	1
8	3	31	12	54	3
9	7	32	4	55	4
10	6	33	7	56	8
11	10	34	3	57	6
12	4	35	3	58	6
13	6	36	7	59	2
14	8	37	2	60	5
15	4	38	8	61	5
16	10	39	9	62	10
17	3	40	2	63	7
18	5	41	4	64	8
19	7	42	6	65	6
20	2	43	4	66	6
21	7	44	3	67	7
22	1	45	6	68	5
23	5	46	7	69	4
				70	5

Annex C (informative) Sample Calculation (cont'd)

The time to background stabilization is five hours and shown by Figure 10. The time the background was measured is

$$t_b = 90 \text{ hours} - 5 \text{ hours} = 85 \text{ hours}$$

NOTE The geometrical or physical characteristics of some samples can increase the time required for return to stabilization as described in section 5. Only data obtained after stabilization should be used to calculate results.

and the sum of the counts B over this time interval is:

B = 344 counts.

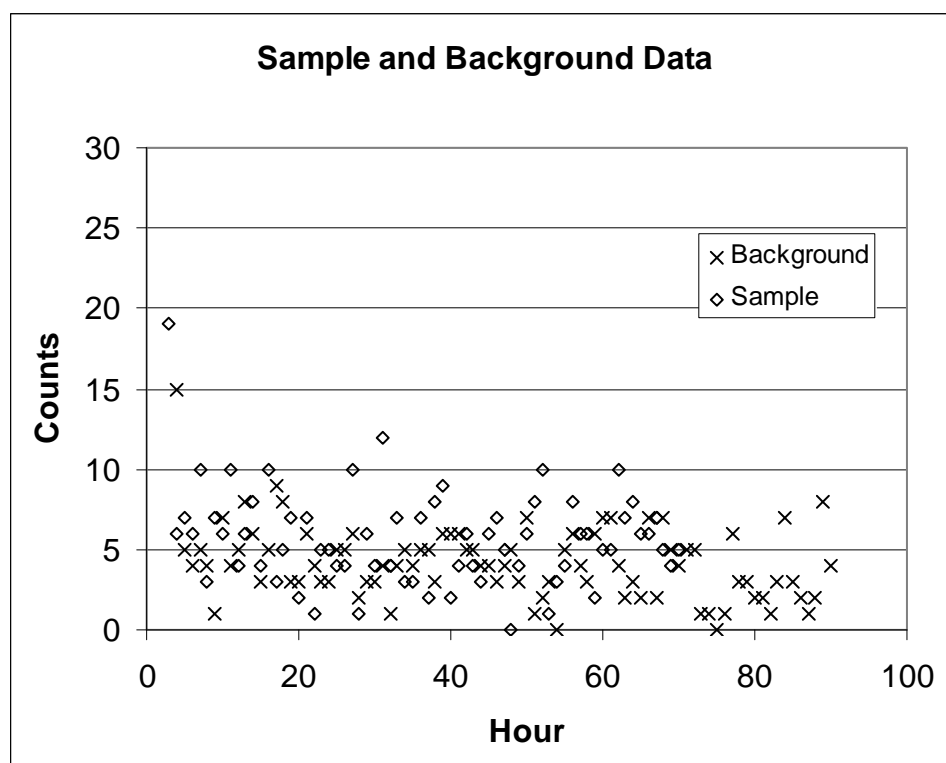


Figure 10 — Example background and sample data

The average background counts per hour is then:

$$344/85 = 4.05$$

The sample counting time is:

$$t_g = 70 - 5 = 65 \text{ hours}$$

and the gross counts (sample + background) are:

$$G = 356 \text{ counts.}$$

Annex C (informative) Sample Calculation (cont'd)

The average sample + background counts per hour is: $356/65 = 5.48$

and the average sample counts per hour S is calculated as: $S = 5.48 - 4.05 = 1.43$ counts per hour.

Given a sample area of 900 cm^2 and a detector efficiency of 90%, the sample alpha emissivity is then determined as

$$\alpha = S(\text{counts/hour})/(\text{Sample area} \cdot \text{Detector efficiency}) = 1.43 \cdot 1000 \text{ hr/khr} / (900 \text{ cm}^2 \cdot 0.9) = 1.8 \alpha \cdot \text{khr}^{-1} \cdot \text{cm}^{-2}$$

and the standard deviation σ calculated as

$$\sigma = \frac{\sqrt{\frac{G}{t_g^2} + \frac{B}{t_b^2}}}{A\varepsilon} = 0.363 \cdot 1000 \text{ hr/khr} / 0.9 \cdot 900 \text{ cm}^2 = 0.448 \alpha \cdot \text{khr}^{-1} \cdot \text{cm}^{-2}$$

The limit of detection at 99.7% confidence is

$$\text{LOD} = 3\sigma = 1.42$$

Or for 90% confidence is

$$\text{LOD} = 1.64\sigma = 0.78.$$



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