APPENDIX C TO PART 61—QUALITY ASSURANCE PROCEDURES

Procedure 1—Determination of Adequate Chromatographic Peak Resolution

In this method of dealing with resolution, the extent to which one chromatographic peak overlaps another is determined.

For convenience, consider the range of the elution curve of each compound as running from -2σ to $+2\sigma$. This range is used in other resolution criteria, and it contains 95.45 percent of the area of a normal curve. If two peaks are separated by a known distance, b, one can determine the fraction of the area of one curve that lies within the range of the other. The extent to which the elution curve of a contaminant compound overlaps the curve of a compound that is under analysis is found by integrating the contaminant curve over the limits $b-2\sigma_s$ to $b+2\sigma_s$, where σ_s is the standard deviation of the sample curve.

This calculation can be simplified in several ways. Overlap can be determined for curves of unit area; then actual areas can be introduced. Desired integration can be resolved into two integrals of the normal distribution function for which there are convenient calculation programs and tables. An example would be Program 15 in Texas Instruments Program Manual ST1, 1975, Texas Instruments, Inc., Dallas, Texas 75222.

$$\frac{1}{\sqrt{2\Pi}\sigma_c}\int_{b-2\sigma_s}^{b+2\sigma_s} e^{\left(\frac{-t_c^2}{2\sigma_c}\right)} dt = \frac{1}{\sqrt{2\Pi}}\int_{\frac{b-2\sigma_s}{\sigma_c}}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx - \frac{1}{\sqrt{2\Pi}}\int_{\frac{b+2\sigma_s}{\sigma_c}}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx$$

The following calculation steps are required:*

1. $2\sigma_s = t_s/\sqrt{2 \ln 2}$
2. $\sigma_c = t_c/2\sqrt{2 \ln 2}$
3. $x_1 = (b-2\sigma_s)/\sigma_c$
4. $x_2 = (b+2\sigma_s)/\sigma_c$
5. $Q(x_1) = \frac{1}{\sqrt{2\Pi}}\int_{x_1}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx$
6. $Q(x_2) = \frac{1}{\sqrt{2\Pi}}\int_{x_2}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx$
7. $I_o = Q(x_1) - Q(x_2)$
8. $A_o = I_o A_c/A_s$
9. Percentage overlap = $A_o \times 100$,

where:

A_s = Area of the sample peak of interest determined by electronic integration or by the formula $A_s = h_s t_s$.

A_c = Area of the contaminant peak, determined in the same manner as A_s.

b = Distance on the chromatographic chart that separates the maxima of the two peaks.

H_s = Peak height of the sample compound of interest, measured from the average value of the baseline to the maximum of the curve.

t_s = Width of sample peak of interest at 1/2 peak height.

t_c = Width of the contaminant peak at 1/2 of peak height.

σ_s = Standard deviation of the sample compound of interest elution curve.

σ_c = Standard deviation of the contaminant elution curve.

$Q(x_1)$ = Integral of the normal distribution function from x_1 to infinity.

$Q(x_2)$ = Integral of the normal distribution function from x_2 to infinity.

I_o = Overlap integral.

A_o = Area overlap fraction.

*In most instances, $Q(x_2)$ is very small and may be neglected.

In judging the suitability of alternate GC columns or the effects of altering chromatographic conditions, one can employ the area overlap as the resolution parameter with a specific maximum permissible value.

The use of Gaussian functions to describe chromatographic elution curves is widespread. However, some elution curves are highly asymmetric. In cases where the sample peak is followed by a contaminant that has a leading edge that rises sharply but the curve then tails off, it may be possible to define an effective width for t_c as ''twice the distance from the leading edge to a perpendicular line through the maxim of the contaminant curve, measured along a perpendicular bisection of that line.''

Procedure 2—Procedure for Field Auditing GC Analysis

Responsibilities of audit supervisor and analyst at the source sampling site include the following:

A. The audit supervisor verifies that audit cylinders are stored in a safe location both before and after the audit to prevent vandalism.

B. At the beginning and conclusion of the audit, the analyst records each cylinder number and pressure. An audit cylinder is never analyzed when the pressure drops below 200 psi.

C. During the audit, the analyst performs a minimum of two consecutive analyses of each audit cylinder gas. The audit must be conducted to coincide with the analysis of source test samples, normally immediately after GC calibration and prior to sample analyses.

D. At the end of audit analyses, the audit supervisor requests the calculated concentrations from the analyst and compares the results with the actual audit concentrations. If each measured concentration agrees with the respective actual concentration within ±10 percent, he directs the analyst to begin analyzing source samples. Audit supervisor judgment and/or supervisory policy determine action when agreement is not within ±10 percent. When a consistent bias in excess of 10 percent is found, it may be possible to proceed with the sample analysis, with a corrective factor to be applied to the results at a later time. However, every attempt should be made to locate the cause of the discrepancy, as it may be misleading. The audit supervisor records each cylinder number, cylinder pressure (at the end of the audit), and all calculated concentrations. The individual being audited must not under any circumstance be told actual audit concentrations until calculated concentrations have been submitted to the audit supervisor.

FIELD AUDIT REPORT

Part A— To be filled out by organization supplying audit cylinders.

1. Organization supplying audit sample(s) and shipping address

____

2. Audit supervisor, organization, and phone number

____

____

3. Shipping instructions: Name, Address, Attention

____

____

____

4. Guaranteed arrival date for cylinders ____

5. Planned shipping date for cylinders ______

6. Details on audit cylinders from last analysis

	Low conc.	High conc.
a. Date of last analysis	....	....
b. Cylinder number	....	....
c. Cylinder pressure, psi	....	....
d. Audit gas(es)/balance gas	....	....
e. Audit gas(es), ppm	....	....
f. Cylinder construction	....	....

Part B—To be filled out by audit supervisor.

1. Process sampled ________________________

____

2. Audit location __________________________

____

3. Name of individual audit _________________

4. Audit date _____________________________

5. Audit results:

	Low conc. cylinder	High conc. cylinder
a. Cylinder number	....	....
b. Cylinder pressure before audit, psi	....	....
c. Cylinder pressure after audit, psi	....	....
d. Measured concentration, ppm Injection #1* Injection #2* Average	....	....
e. Actual audit concentration, ppm (Part A, 6e)	....	....
f. Audit accuracy:[1]		
Low Conc. Cylinder	....	....
High Conc. Cylinder	....	....
Percent [1] accuracy= $\frac{\text{Measured Conc.} - \text{Actual Conc.}}{\text{Actual Conc.}} \times 100$		
g. Problems detected (if any)	....	....

[1] Results of two consecutive injections that meet the sample analysis criteria of the test method.

[47 FR 39178, Sept. 7, 1982]

APPENDIX D TO PART 61—METHODS FOR ESTIMATING RADIONUCLIDE EMISSIONS

1. Purpose and Background

Facility owners or operators may estimate radionuclide emissions to the atmosphere for dose calculations instead of measuring emissions. Particulate emissions from mill tailings piles should be estimated using the procedures listed in reference re #2. All other emissions may be estimated by using the