UNCLASSIFIED		DOCUMENT NO. ARH-SA-104	
CLASSIFICATION		DATE 10-15-71	COPY NO. CONF 71171-14
ISSUING FILE	Atlantic Richfield Hanford Company RICHLAND, WASHINGTON		
	MASTER		
TITLE AND AUTHOR ENVIRONMENTAL LABORATORY QUALITY ASSURANCE - D. J. Rochon			
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ROUGH DRAFT

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October 15, 1971

ARH-SA-104

CONF-71111--4

ENVIRONMENTAL LABORATORY QUALITY ASSURANCE

The product of an analytical laboratory is a measurement generated for a customer who is concerned with the quality of that measurement. It is the purpose of the laboratory quality control program to make the quality of the measurement what it should be, and the purpose of the quality assurance program is to make sure the quality of the measurement is what it should be.

Since an analytical laboratory generally performs analyses on only a portion of the total material, the quality assurance program begins with a quality control program for sampling. The validity of the analytical measurement rests on the sample being representative of the total material.

For the radioisotopic analysis of environmental samples, it is essential that the laboratory know how the sample was taken so that they may apply an appropriate analytical procedure for the analysis required of that sample. In selecting an appropriate container for aqueous solutions containing radionuclides at concentrations of 1×10^{-4} uci/l and less, many factors must be considered to minimize adsorption effects on the vessel walls. These factors include the pH of the solution, the vessel material, and the chemical composition and concentration of the solution. Unfortunately, the dependency on the factors mentioned is not the same for different elements, and for others it has not been adequately studied. For a complex mixture of different radionuclides

having different valency states requiring chemical separations, the particular scheme of analysis to be performed should be designed to minimize all effects.

Once the sample has been taken and an analysis scheme has been decided upon, the next step is a quality control program to determine independently that both the analytical method and the instrumentation used are in control. A series of tests is performed on each instrument at pre-determined frequencies to guarantee the integrity of the analysis. The instrument control tests also alert personnel to off-standard conditions for which maintenance may be planned and scheduled.

In order to control the analytical method, either or both radioactive tracers and synthetic standards that approximate sample solutions are used. The frequency of standards analyses is predetermined such that a specified number of analyses for a given method will be distributed throughout the month over all analysis periods. At ARHCO, an IBM 1800 process control computer has been programmed to generate an analysis card corresponding with the predetermined frequency, to calculate the results of the analysis, to compare the current result with a set of previously generated standards data, and to indicate to the generating laboratory whether the method has changed significantly in either the average recovery or the precision.

Since the values of the control standards must be known by the using laboratory in order to control the conditions, and since the knowledge

that an analysis is being performed on a standard ^Amy subconsciously influence the manner in which the laboratorian performs the analysis. We use a synthetic standard prepared to be a close approximation of the sample which is submitted to the laboratory and identified as a sample in order to determine the precision and accuracy that may be expected from that analysis on a routine basis. It is economically impractical to provide synthetic sample standards for every sample type. However, an additional mechanism which can provide at least the same information as well as the interaction between different laboratories, is the participation in sample exchange programs, such as the exchange program conducted by the International Atomic Energy Agency.

The quality assurance program in its entirety may be represented as different levels in a system of concentric circles with the sampling control program at the locus. (See Figure I, page .) The total program is synergistic in the sense that the effect of applying each succeeding level is greater than the sum of prior effects taken independently.

Figure I

Inverse of Quality Assurance Program

The synergistic effect is best illustrated by examples. First, let us consider the synergism between the sampling and analytical levels. A range of a factor of 10 was observed for the results of the alpha activity in aqueous process condensate streams. The nominal activity observed was 5×10^{-4} uci/l. Standards for both the method and the instrument indicated a variation of $\pm 20\%$ would be the maximum expected for a static operating condition. It was noted that the samples received for analysis were in either polyethylene or glass containers, whereas the standards were always submitted in polyethylene containers. A series of standards in glass containers ~~were~~^{used} issued to the laboratory for alpha analysis. The results of this test revealed that both plutonium and americium, which are the most probable alpha emitting isotopes to be found in that particular stream, selectively adsorbed onto glass. The difference in the recovery of the standards submitted in glass and the recovery of the standards in polyethylene accounted for the factor of ~~10~~ range observed in samples.

As an example of the intralaboratory level, let us consider the determination of strontium-90, which involves a chemical separation with subsequent determination of the beta activity. Both the method and the instrument control standards indicated statistical control conditions were being obeyed. The statistical analysis of a number of standards with different concentration indicated the analysis was significantly less precise than would be expected from the precision of the control standards. Further analysis of the data indicated that the standard recovery varied with

the activity of the sample mount. As a result of this investigation, it was discovered that the instrument calibration for efficiency was mount rate dependent. As long as the activity on the sample mount was within $\pm 50\%$ of the activity of the mount used to determine the instrument efficiency, the precision of the standards analyzed was not significantly different from the precision as determined by the control standards. However, when the activity of the mount exceeded the activity of the instrument efficiency standard by 100%, the standard recoveries were biased low.

As an example of the interlaboratory level synergism, let us consider the analysis cesium-137. Laboratory A utilizes two independent methods in the analysis of this isotope. One method involves the chemical separation of cesium with a subsequent determination of the activity by gamma energy analysis. The second method is a gamma energy analysis of a direct mount of the sample without preliminary separations. Laboratories B and C determine the activity without chemical separation. Aliquots of the same material were analyzed in quadruplicate by all three laboratories with the results tabulated in Table I-a. Each of the three laboratories' control data again indicated statistical control conditions were obeyed. The discrepancy between the two independent methods of analysis at Laboratory A was found to be due to using improper calibration parameters. Two different calibrations exist for the direct mount technique; one is for mounting on absorbent pads and one is for a dried mount. The samples were mounted on the absorbent pads and the

activity calculated from the dry mount calibration. Further investigation revealed that the discrepancy between laboratories resulted from a variation in the decay scheme value for the electron conversion. Assuming the same decay scheme, the results in Table I-b now show no statistically significant differences between the averages.

Laboratory Method			
	A	B	C
Direct	.964X $\pm$.006	1.001X $\pm$.003	.948X $\pm$.003
Separation	.927X $\pm$.035		

Table I-a

Laboratory Method			
	A	B	C
Direct	1.003X $\pm$.006	1.001X $\pm$.003	.998X $\pm$.003
Separation	.990X $\pm$.035		

Table I-b

Table I Comparison of laboratory standards data on ^{137}Cs with X being the standard value a) before data corrections applied and b) after corrections applied.

No data is as yet available for the analysis of the same environmental sample aliquot analyzed at different locations. ARHCO laboratories have been involved in a number of different types of sample exchanges with other locations. Participation in these exchange programs has been of inestimable value, so there is no reason to expect any different results from participation in exchange programs dealing with environmental samples.

From the examples presented, it can be seen that in order to make sure the quality of the measurement is what it should be, it is not sufficient to have only a control program instituted; it is necessary to have a program in place which will continuously examine the documentation available for possible discrepancies which can be corrected before results are issued which may be challenged.