APPENDIX B

ANALYTICAL PROCEDURES FOR CHEMICALS

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ABSTRACT

This appendix summarizes the various methods that were used by the Savannah River Site (SRS) laboratories to analyze samples for nonradiological or chemical contaminant concentrations. Methods of analysis used by organizations other than the SRS are not provided.

MERCURY

A variety of units was used for reporting mercury concentrations in environmental samples. In general, sediment and soil concentrations were reported as parts per million (ppm), parts per billion (ppb), or µg Mercury g^{-1} soil dry weight. Water concentrations were reported as ppm, ppb, or µg Mercury l^{-1} water. Fish concentrations were reported as ppm, ppb, or µg Mercury g^{-1} flesh wet weight. A May 1971 report (Du Pont 1971a) described a procedure for analysis of mercury in environmental samples by the SRS. Water samples were buffered at pH 6.0 ± 0.5, 200 mg of mercury-free cadmium sulfide were added, and the slurry was mixed for 15 minutes. The sulfide and sorbed mercury were filtered on an asbestos pad, which was washed with methyl alcohol to remove organic material and dried overnight at 100°C. The mercury was then distilled at 550°C, and vapor was collected in an absorption cell for atomic absorption spectrophotometry. The lower limit of detection (LLD) in water samples was 0.5 ppb, and analytical precision was about 10 to 15% for 1 to 5 ppb.

A June 24, 1971 letter from W.P. Bebbington to N. Stetson (Bebbington 1971) indicated the LLD in water samples analyzed in 1970 to be 2 ppb for the routine method, which was not described. However, a special and tedious method not practical for routine monitoring, which also was not described, appeared to have a LLD of 0.1 ppb, based on tabulated data.

A new method for mercury analysis of environmental samples (flameless atomic absorption spectrophotometry) was described in a Savannah River Laboratory September 1971 report (Du Pont 1971c). Sediment samples were prepared by oxidation with $KMnO_4$ followed by leaching with concentrated HCl or H_2SO_4. The mercury was then reduced to elemental Mercury with $SnCl_2$. Air was bubbled through the solution, which carried Mercury vapor through the absorption cell of a Perkin-Elmer Model 403 atomic absorption spectrophotometer. Quantitative assessment of mercury concentrations were then made based on the measured absorption of the 2537 Å line. Tracer studies showed that $H_2SO_4/KMnO_4$ digestion recovers 96% of the mercury. The LLD for sediment samples was not provided, but a 1974 memorandum (Horton 1974) indicated a LLD of 2 ppb for the same method of analysis. This was considered sufficient since background levels in soil and streambed sediments vary from 20 to 50 ppb. A LLD of 30 ppb was reported for a study conducted on stream and river sediment in 1973 (Gladden et al. 1985).

Water samples were analyzed following overnight digestion with H_2SO_4 and $KMnO_4$ at room temperature. Fish samples were dissolved in concentrated H_2SO_4 at 55° C, and organic material was oxidized by $KMnO_4$ at room temperature. Mercury concentrations in the resulting solution were determined as described for sediment. The LLD for water samples was reported as 0.05 ppb with an analytical precision of about 10% at 0.1 ppb. The LLD for fish samples was not

given but analytical precision was reported as 12% for 500 ppb. An August 1971 document (Du Pont 1971b) reported a LLD of 10 ppb using flameless atomic absorption spectrophotometry with a Coleman mercury analyzer system. Mercury measurements for all environmental media appear to have been made using standard flameless atomic absorption spectrophotometry as described by the Environmental Protection Agency (EPA) (EPA 1983) since 1971.

CHROMIUM

Methods of analysis for chromium and other metals were not explicitly described in the environmental monitoring reports that have been reviewed. The annual report for 1983 (Ashley et al. 1984) indicated that a large part of the nonradioactive analyses was performed by an offsite vendor (Envirodyne Engineers, Inc. was the vendor in 1983). Standard EPA methods of analysis (EPA 1983), such as atomic absorption spectroscopy for chromium, were likely used for these analyses.

OTHER CHEMICALS

Other chemical analyses were carried out using standard EPA methods of analysis, many of which are described in EPA (1983).

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