

INFORMATION FOR
NUCLEAR REGULATORY COMMISSION

REGARDING
UNITED NUCLEAR CORPORATION'S
(WOODRIVER JUNCTION, RHODE ISLAND)
WATER SOLUBILITY TEST PERFORMED ON SOIL

PREPARED BY
JAMES J. MUELLER

JULY 14, 1982



Controls for Environmental Pollution, Inc.
Santa Fe, New Mexico 87502

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Introduction

This document has been prepared by Controls for Environmental Pollution, Inc., (CEP), as requested by Karl Helgeson of United Nuclear Corporation, Wood River Junction, Rhode Island.

The following discussion will include; analytical methodologies used to analyze United Nuclear Corporation's water solubility test samples.

1.0 Solubility Procedure

The procedure used to water leach the soil is the same rotating method described in ASTM D 19:12 Method and approved by the U.S. Environmental Protection Agency for performing leach studies for hazardous waste analyses.

The following is the procedure used for the solubility tests:

1.1 Apparatus

1.1.1 500 ml wide-mouth polyethylene bottle with tightly fitting screw cap.

1.1.2 A rotating apparatus consisting of a box with six (6) side-by-side compartments each of which will hold one of the above bottles; so arranged that when the motor is started, the bottles are rotated end-over-end about forty times per minute.

1.2 Procedure

1.2.1 Weigh out 50 grams of dry soil into a 500 ml wide-mouth plastic bottle.

1.2.2 Add 200 ml D.I. water and shake for a minute to mix.

1.2.3 Place tightly capped bottle in a compartment of the rotator, fasten lid, and start rotation.

1.2.4 After twenty-four hours rotation, remove bottle from rotator and filter entire sample.

2.0 Analytical Methods

The procedures used for this contract are routinely utilized by CEP in the analyses of environmental samples. The analytical methods used have been studied collaboratively and found acceptable. CEP's analytical procedures are the same as, equal to, or better than those currently used by the Environmental Protection Agency. All data reported by CEP and the limit of sensitivity to each analytical measurement technique is reported at the 95% confidence level. These methods are derived from, "EML Procedure Manual", HASL-300, U.S. Department of Energy, New York, N.Y., U.S. Environmental Protection Agency "Radiochemical Method Manual", Los Alamos Scientific Laboratory "Radiochemical Procedures", LA-1721 and J.J. Mueller, "Radio and Water Chemistry Manual", June 1966, ACNP-66538.

2.1 Filtered Liquid from Solubility Test

2.1.1 Gross Alpha and Beta (Total)

An aliquot of the filtered liquid was evaporated to dryness and transferred to a weighed planchet. The Gross Alpha and Gross Beta radioactivity was measured by counting the planchet in an internal gas flow proportional low background counter (Beckman Wide Beta II) which has a sixty percent beta efficiency using Strontium, Yttrium-90 and forty percent alpha efficiency using Plutonium-239. The self-absorption correction was performed on all samples which is based on mg/cm^2 of dissolved and suspended solids collected on planchet.

2.1.2 Radium-228, Radium-226 (Sequential Method)

Appropriate aliquots of filtered liquid were transferred to beakers. The Radium-228 and Radium-226 in a water sample were concentrated and separated by coprecipitation with barium and lead as sulfates and purified by EDTA-Chelation. After a thirty-six hour ingrowth of

Actinium-228 from Radium-228, the Actinium-228 was carried on Yttrium Oxalate, purified and beta counted. The Radium-226 in the supernate was precipitated as the sulfate, purified and alpha counted utilizing an alpha spectrometer.

2.1.3 Strontium-90 (14 Day Ingrowth Method)

An aliquot of the filtered liquid containing standardized Strontium and Yttrium carriers, was stirred with Dowex 50W-X8 cation exchange resin at a pH of 6 for thirty minutes. All nuclides were stripped from the resin with strong acid. After the ingrowth period has been established, the Yttrium-90 was extracted with five percent di-2-ethylhexyl phosphoric acid (D₂EHPA) in toluene, back extracted into an aqueous phase, precipitated as the oxalate and counted in a low background internal gas flow proportional counter (Beckman Low Beta I) to determine the Strontium-90 content of the sample. The system has a Strontium, Yttrium-90 efficiency of sixty percent.

2.1.4 Isotopic Uranium (U-234, U-235, U-238)

A suitable aliquot was spiked with a standardized Uranium-232 solution. The sample was purified by passing the solution through an ion exchange resin column. The eluate was evaporated and electroplated onto a stainless steel disc. The counting of the sample was performed on a surface barrier detector utilizing a multichannel analyzer (Northern Scientific). The Uranium recovery was determined from the Uranium-232 spike recovery.

2.1.5 Isotopic Thorium (Th-228, Th-230, Th-232)

Appropriate duplicate aliquots of the filtered liquid were transferred to a beaker. One aliquot serves as the sample and the other was spiked

with a standard Thorium-232 solution. The sample and spike were purified by passing the solution through an anion exchange column. The eluate from each was evaporated and electroplated onto a stainless steel disc. The samples and spikes were counted utilizing a surface barrier detector connected to a multichannel analyzer (Northern Scientific). The Thorium recoveries were determined from the spike aliquots and subsequently the activity of each sample was determined.

2.1.6 Gamma Spectrometry (Cesium-137 and Other Nuclides)

The entire 200 ml of filtered liquid was placed in a Marinelli beaker and counted with a multichannel analyzer equipped with a Ge(Li) detector which is coupled to a 4096 channel, computer based, multichannel analyzer (Northern Scientific). The resulting spectrum was analyzed by the computer, and specific nuclides, if present, identified and quantized.

3.0 Leach Soil From Water Solubility Test

3.1 Preparation Method

After the filtration was complete (all water vacuum filtered from soil), the water leached soil was transferred into new, clean drying pans. The drying pans filled with water leached soil were placed into an oven at 100°C and allowed to dry thoroughly.

3.2 Gross Alpha and Beta

A 10-gram aliquot of the water leached soil was ashed in a muffle furnace and dissolved in hydrofluoric-nitric acid mixture. The residue was dissolved in dilute hydrochloric acid and evaporated several times with 8N nitric acid and transferred to a tared planchet. The Gross Alpha and Gross Beta radioactivities were determined using a low background gas proportional counter (Beckman Wide Beta II). The Gross Alpha and Gross Beta activities were corrected for self-absorption.

3.3 Cesium-137, Radium-226 and Radium-228

All the water leached soil, before any analytical analysis started, was placed in a precalibrated plastic bottle and counted for eight hours, on a Ge(Li) detector, which is coupled to a 2048 computer based, multi-channel analyzer (Northern Scientific). The resulting spectrum was fed into a computer and specific nuclides, if present, were identified and quantized in terms of energy and net count rate with the aid of the computer. This method was approved by EPA to determine Radium-226, Radium-228 and Cesium-137 in soil.

3.4 Strontium-90 (14-day Ingrowth Method)

Ten-grams of water leached soil was added to a platinum crucible, and it was ignited at 400-500°C to destroy organic matter. The soil was fused with Na_2CO_3 and mixed thoroughly. The fusion was at 900°C in a muffle furnace until the melt was clear. After cooling, the fused mass was ground to fine powder utilizing a pestle. The fused ground powder was dissolved in hot water and the Strontium-85 tracer was then added.

Slowly, with continuous stirring, add one liter of HCl. Evaporate with mechanical stirring to a paste.

Dilute to 2.5 liters with water and allow the silica to settle.

Filter with suction through a double 24 cm glass fiber filter. Wash with 200-300 ml of hot 1:1 HNO_3 followed by 250 ml of water. Discard the residue.

Transfer the filtrate and washings to the original beaker. Dilute to 1500 ml with water. Add 50 grams of oxalic acid with magnetic stirring until the salt is completely dissolved.

Adjust the pH to 5.5 - 6.0 with NH_4OH . (If the brown color of ferric hydroxide persists, add more oxalic acid and readjust the pH.) The optimum condition is an excess of oxalic acid in solution without causing crystallization of ammonium oxalate upon cooling.

Allow to stand for several hours, stir and filter with suction on a 15 cm #42 Whatman filter paper. Discard the filtrate.

Transfer the precipitate and paper to an appropriate size nickel or platinum dish and dry overnight at 110°C. Ignite the oxalate precipitate in an electric muffle at 400-500°C for 2 hours. Raise the temperature slowly to about 700°C and continue heating for 2 hours. (Spattering is minimized by ignition in this manner.)

Cool the precipitate and transfer to an appropriate size beaker. Dissolve in a minimum of 1:1 HNO₃. Add about 6 drops of H₂O₂ (30%) to facilitate dissolution. Gently heat to boiling.

Cool to room temperature. If insoluble material is present at this point, filter by suction through a double 7 or 9 cm glass fiber filter. Discard the filter and residue.

Transfer the solution to a suitable size beaker and evaporate to dryness. The evaporation must be done slowly to avoid spattering.

Dissolve the salt in water and perform successive fuming nitric acid separations, (the first two separations at concentrations slightly greater than 75%) until the strontium has been separated from the bulk of the calcium. Samples with a high calcium content will require 5 or 6 more separations. The volumes of 75% HNO₃ solutions may be changed as required by the mass of calcium present, keeping in mind that minimum volumes are always best.

The procedure described under section 2.1.5 completes the analysis. The Strontium-85 was used to determine the recovery of the method.

3.5 Isotopic Uranium (U-234, U-235, U-238)

CEP used the following analytical method for analyzing Uranium-234, 235, 238 in soil: A ten-gram aliquot of the water leached soil was spiked with Uranium-232 tracer. Total dissolution of the soil was performed using hydrofluoric-nitric acid mixture, nitrated and evaporated to dryness. The residue was dissolved in concentrated nitric acid and again taken to dryness and redissolved in dilute acid. The sample was purified with an ion exchange resin column. The Uranium was electroplated and the discs counted on a solid state alpha spectrometer and the chemical recovery was determined from the Uranium-232 tracer peak.

3.6 Isotopic Thorium (Th-228, Th-230, Th-232)

CEP used the following analytical method for analyzing Thorium-228, 230, 232 in soil: Two ten-gram aliquots of the water leached soil were taken and Thorium-232 external tracer added to of the aliquots. Total dissolution of the soil was performed using hydrofluoric-nitric acid mixture. The residue was dissolved in dilute hydrochloric acid. The samples were purified with an ion exchange resin column. The Thorium was electroplated and the stainless steel disc was counted on a solid state alpha spectrometer and the chemical recovery was determined from the Throium-232 tracer peak in the second aliquot.

4.0 Other Information

See Document dated June 25, 1982 entitled "**Water and Soil Sample Analyses**" by James J. Mueller, Controls for Enviornmental Pollution, Inc. for United Nuclear Corporation's, Woodriver Junction, Rhode Island Plant for details on calculation methods, instrumentation and other details on Quality Control.