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ANALYSIS OF VEGETATION FOR I^{131}

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by

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ANALYSIS OF VEGETATION FOR I-131

INTRODUCTION:

The operating experience at Hanford Works has indicated that one of the major hazards encountered with the disposal of the gases from the dissolution of the metal is the deposition of I-131 on the vegetation.[1,2] In order to evaluate this hazard with a sensitivity required by more recent stringent tolerances, a procedure for the analysis of I-131 in vegetation was necessary. Previous measurements were performed by counting a one gram sample directly on the second shelf of a mica-window GM counter.[3] The results were corrected by a factor of three for self-absorption of the beta particles in the vegetation and by factor of 12 for the geometry of the counter. This method gave a sensivity estimated at 0.04 μc/kg which was insufficient to detect the proposed new tolerance value of 0.009 μc/kg.

The limitations set on the new method of analysis were that it would be sensitive to about 1/5 or 1/10 of the new tolerance value, and that the method be simple enough so that a large number of analyses could be made in a routine manner without requiring large amounts of laboratory space, or a long processing time. It was obvious that any method which would give satisfactory results must incorporate a procedure of removing the I-131 from the vegetation so that difficulties with self-absorption would not be encountered. This should result in a gain of sensitivity from three factors; a larger sample may be extracted than may be counted directly, the self-absorption of the beta particles by the vegetation is reduced and it is physically possible to count the sample at a higher geometry on the first shelf, instead of on the second shelf.

SUMMARY:

An analytical procedure in which the iodine is extracted from the vegetation by the use of a caustic solution is described. An average yield of 83± 18% is indicated from the spiked samples which have been run to test the procedure while

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yields of about 70% are obtained on routine samples. A limiting sensitivity of 0.002 to 0.004 μc/kg results when a 5 gm. sample is analyzed. The amount of time consumed in the analysis is greater than for the direct mounting procedure but is sufficiently small so that large numbers of samples may be run.

PROCEDURES:

Several procedures were tried before the most promising one was chosen and efforts concentrated on refining it to the point where it could be used. Initially, an alkaline digestion was used to extract the I-131 away from the vegetation. This extract was neutralized, filtered, and reduced and an acid oxidation with sodium nitrite was then used to convert the I-131 from iodide ion to elementary iodine. A chloroform extraction was made and the chloroform layer reextracted with an aqueous reducing solution of sodium bisulfite. Excess reducing agent was destroyed by boiling and the I-131 was precipitated as silver iodide. This precipitate was filtered, dried, mounted and counted on a mica-window counter. Since difficulties were encountered with the emulsification of the chloroform layer, sodium bisulfite was substituted as the extractant. Some emulsification still occurred and an attempt was made to carry out the chloroform extraction by refluxing, instead of shaking in the separatory funnel.

An attempt was made to digest the vegetation in hot 8 N nitric acid with provisions to scrub the vapors evolved with dilute alkali. This was carried out by distilling the contents of the acid digestion flask to dryness while continually condensing and neutralizing the distillate. The neutralized distillate plus the scrubber solution were reduced and silver iodide precipitations were made. It was hoped that by this procedure the same samples could be used for the I-131 analysis and for analysis of other beta emitters which may have been deposited.

The procedure finally adopted consists of an alkaline digestion of the vegetation using mixed sodium-hydroxide and sodium bisulfite. After filtering, the dissolved organic matter is largely removed by an aluminum hydroxide precipita-

tion, and the iodine is precipitated from an acid solution with silver nitrate. Thus, emulsion formation is avoided and absorption of beta particles by organic matter in the final precipitate is minimized. The complete procedure is given in the appendix.

All samples were counted on the first shelf of a mica-window counter with a window thickness of 2-4 mg/cm^2. Geometry corrections, including a correction for extended rather than point source, have been applied as well as backscatter corrections. No self-absorption correction has been made to these data. All values are corrected to a common date by the use of the eight day half-life of I-131. The yields were evaluated by adding a known amount of I-131 to the sample before analysis. This amount of I-131 was measured by placing aliquots of the solution used on a stainless steel counting plate and precipitating silver iodide by the addition of a small amount of silver nitrate, the plates were then dried under an infra-red lamp for a minimum time and counted.

RESULTS:

The first procedure tried, utilizing a chloroform extraction with sodium bisulfite as an initial extracting agent, gave yields of the order of 50-70%. The difficulties encountered with emulsification, however, were rather undesirable. A yield of about 16% was obtained when the chloroform extraction was performed by refluxing instead of shaking in a separatory funnel.

The procedure involving digestion of the vegetation in hot nitric acid with consequent scrubbing of the vapors gave low yields of 33 and 12% respectively, believed to have been caused by absorption of I-131 on the charred residue remaining after acid digestion. Since this procedure would require large amounts of equipment in order to carry the digestion to completeness, it was abandoned.

Table I gives the results of 16 analyses performed on five 5-gram samples of vegetation by the final procedure as listed in the appendix.

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Table I

Analyses of 16 Spiked Vegetation Samples

Sample Number	Spike Activity D/M	D/M Extracted	% Yield
8-25	2730	2520	92
8-27	2300	2121	92
8-30	1775	1653	93
9-2(a)	1370	1046	76
9-2(b)	1370	853	62
9-4(a)	1150	1013	88
9-4(b)	1150	972	85
9-8	815	683	84
9-9	750	667	89
9-10	685	533	78
9-14	485	419	86
9-15	445	345	77
9-17	373	336	90
10-1	110	75	68
10-5	72.5	46.2	64
10-6	66.5	65.9	99

Average yield = 83%
Std. Dev. of yield = 11%

The overall yield and the reproducibility of the method appears quite satisfactory especially as these were the first samples to be run by this particular procedure. A decay curve measured with the iodine extracted from vegetation collected near the separations areas followed the eight day half-life of I^{131} over a period of four half-lives indicating that little of the other fission products known to be present in the vegetation have been extracted.

The performance of the procedure for routine analyses is illustrated in Figure 1. The upper line on this graph represents the daily measurements of 0.1 ml of the spike solution spread over a 1" diameter. The points below the line represent the counting rates obtained on the filter papers from the analysis of five grams of vegetation spiked with 0.1 ml of the I^{131} solution.

The average ratio of these two counting rates for 62 samples is 0.37 with a standard deviation of 0.12. In order to obtain an estimate of the chemical yield the counting rate of the "spike" solution should be corrected by a factor of 1.05

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for the 1/2" diameter of the source, 1/1.26 for backscatter from the stainless steel plate; and the counting rate of the sample should be corrected by 1.64 for the 1-1/2" diameter of the source and 1/1.10 for backscatter from the filter paper[4]. This gives a value of 67±(20)% yield for the extraction which is probably lower than actual because of self-absorption in the residue on the paper. The wide variation is undoubtedly due to differences in self-absorption from various amounts of crud carried down from changes in type of sample.

The average counting rate above background for 63 blank samples analyzed according to this method was 0.4±(1.2) c/m at 23% geometry.

DISCUSSION:

The principal source of difficulty in extracting the I^{131} from vegetation is the organic matter which extracts with it. Most of this material is removed by the aluminum hydroxide precipitation. However, any that remains will tend to interfere in the silver iodide precipitation. Various types of vegetation appear to vary markedly in their ability to interfere. Green rabbit bush and dry cheat grass appear to be the worst offenders, but even these materials are handled successfully on a routine basis.

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2. Singlevich, W. HW 13743. June 22, 1949
3. Healy, J. W. and Panke, L. D. HW 3-3598. August 1, 1946
4. Schwendiman, L. C. Unpublished Report

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APPENDIX

EXTRACTION OF I-131 FROM VEGETATION

The following vegetation extraction has been developed to increase the sensitivity of detection of I-131 deposited on vegetation.

Equipment	Chemicals
500 ml. beaker	Iodide carrier solution (10 mg. I^- per ml.)
Stirring rod	1.0N sodium bisulfite
# 0 Buchner funnel	0.2N sodium hydroxide
250 ml. filter flask	1.0N aluminum nitrate
# 2 Buchner funnel	8.0N nitric acid
90 ml. Lusteroid centrifuge tubes	0.1N silver nitrate
Hot plate	
100 ml. graduated cylinder	
25 ml. graduated cylinder	
Alk-Acid test paper	
Transfer droppers	
8.0 cm asbestos filter discs	
4.25 cm paper filter discs	

PROCEDURE

1. Add 5 grams of vegetation to 100 ml. of a solution which is 0.2M in sodium hydroxide, 0.01M in sodium bisulfite and 0.00158M in sodium iodide.
2. Heat the mexture to boiling (about 3 minutes) and filter through an asbestos filter paper using a few milligrams of filter aid. (Hyflo Super Cell).
3. Boil the filtrate for 20 minutes.
4. Add 20 ml. of 1.0N aluminum nitrate solution, with stirring.
5. After cooling, determine the pH of the mixture (using Alk-Acid Test paper) and adjust to pH = 6.0, using dilute nitric acid or sodium hydroxide as required.
6. Centrifuge the sample and discard the aluminum hydroxide precipitate.
7. Adjust the pH of the supernatant to 2.0 by adding dilute nitric acid.
8. Add 10.0 ml. of 1.0N silver nitrate and heat nearly to boiling.
9. Filter the silver iodide precipitate using No. 5 paper and wash with a small amount of alcohol.
10. Dry at room temperature, mount the filter paper on a sample card and count.

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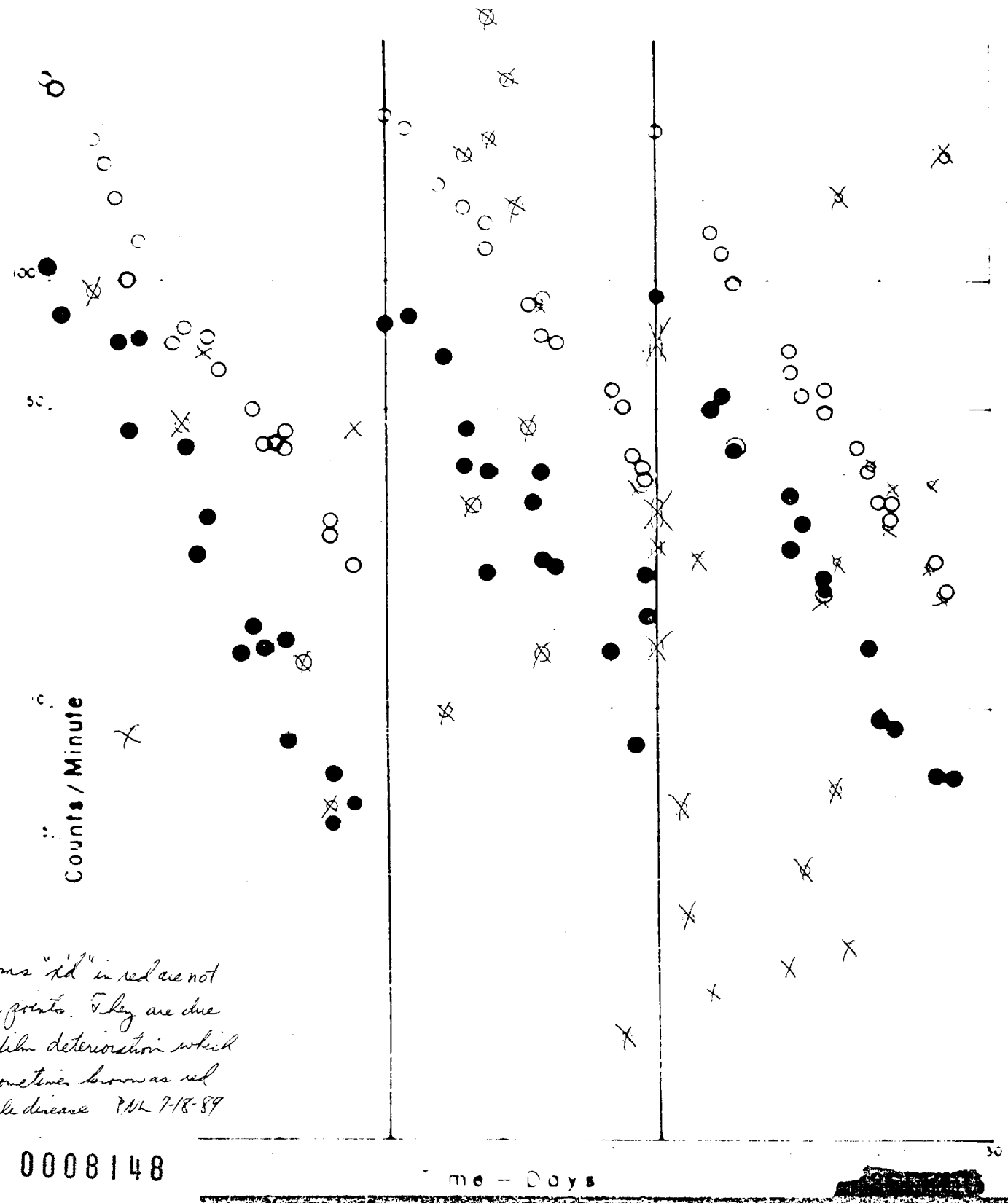
FIGURE 1

YIELD OF SPIKED VEGETATION SAMPLES

○ – Counting Rate of I^{131} Added

● – Counting Rate of Analyzed Sample



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