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TITLE

THE SEQUENTIAL SEPARATION AND DETERMINATION OF ARSENIC-76 AND PHOSPHOROUS-32 IN REACTOR EFFLUENT WATER

MASTER

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THE SEQUENTIAL SEPARATION AND DETERMINATION OF ARSENIC-76 AND PHOSPHOROUS-32 IN REACTOR EFFLUENT WATER

by

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CHEMICAL PROCESSING DEPARTMENT
GENERAL ELECTRIC COMPANY
RICHLAND, WASHINGTON

June 12, 1962

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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THE SEQUENTIAL SEPARATION AND DETERMINATION OF ARSENIC-76 AND PHOSPHOROUS-32 IN REACTOR EFFLUENT WATER

ABSTRACT

A procedure is described for the sequential separation and determination of Arsenic-76 and Phosphorous-32 in reactor effluent water. The analysis can be performed in less than one hour and the arsenic fraction is scanned immediately after separation on a multi-channel gamma spectrometer. The discrete gamma energy of .56 MEV[4] for Arsenic-76 makes further purification unnecessary.

The organic phase containing the Phosphorous-32 is dry mounted on a one-inch stainless steel dish for beta counting.

THE SEQUENTIAL SEPARATION AND DETERMINATION OF ARSENIC-76 AND PHOSPHOROUS-32 IN REACTOR EFFLUENT WATER

I. INTRODUCTION

Disposal of reactor effluent water into the Columbia River requires that periodic measurements be made of the radioisotopes present. These measurements are used to determine results of tests being run at the reactors and for historical information.

In the spring of 1961, the Atomic Energy Commission requested that steps be taken to reduce the phosphorous-32 content in reactor effluent water. Although the phosphorous-32 concentration was considerably under the maximum permissible limits, even lower levels were desired. In an attempt to fulfill the request, a high-alum concentration treatment of the water was started.

Previous analytical data had shown that a specific ratio exists between arsenic-76 and phosphorous-32 in the reactor effluent water.[1] Therefore, by monitoring the arsenic-76 on a day-to-day basis, effective control of the alum concentration could be maintained and rapid information on phosphorous-32 could be developed. Current methods used to determine arsenic-76 did not meet the control requirements of the proposed test; therefore, a method for rapid and accurate determination of arsenic-76 was necessary.

II. EQUIPMENT AND REAGENTS

A. Wrist shaker

B. Transistorized multi-channel gamma analyzer

C. Beta Proportional Counter

D. CP nitric and hydrochloric acids

E. Ten per cent (10%) solution of ammonium molybdate

F. CP Butanol

G. CP Chloroform

H. CP Ethyl Acetate

III. PROCEDURE

A 50 ml aliquot of effluent water is adjusted to a pH of 7-8 with dilute NH_4OH or HNO_3. Then, 6 ml of a 10% solution of NH_4MoO_4 is added followed by a two-minute standing period. The molarity is adjusted to 1.3 using 8 MHNO_3 followed by the addition of 5 ml butanol. The phosphorous is extracted using 15 ml of 1:3 mixture of butanol and chloroform, then rinsing with 5 ml of the same mixture and combining the organic phases.

The organic phase is evaporated to 3 ml in a small beaker and transferred to a one-inch stainless steel counting dish, evaporated to dryness and beta counted for phosphorous-32.

To the remaining aqueous phase, add 15 ml of a 1:1 mixture of ethyl acetate and butanol and shake for one minute. Add 15 ml of chloroform and shake again. The aqueous phase is discarded and the organic phase rinsed with 10 ml of 8NHCl. Then, 20 ml of the organic is placed in a vial for gamma analysis of arsenic-76.

IV. DISCUSSION

A. Phosphorous and Arsenic Separation

1. From this separation arsenic-76, the major interference of phosphorous-32, is completely eliminated. (See figure I.)

2. Time required for analysis is less than one hour.

B. Decontamination

1. Separation of arsenic-76 from other major interfering radioisotopes in reactor effluent water is adequate. These include Na^{24}, Mn^{56}, Cr^{51}, Cu^{64}, Np^{239}, and to a lesser extent La^{140}. (See figure II and III.)

2. Interferences from other radioisotopes common to the sample. (See figure IV.)

3. Gamma analysis for arsenic is much more specific than beta counting. The spectrum can be viewed directly on the gamma analyzer oscilloscope or printed on tape; thus, enabling positive identification of the radioisotope (s) present.

4. One can see from the gamma spectrum that the arsenic would be difficult to calculate without a separation. (See figure II and III.)

C. Method Efficiency

1.

Volume Organic / Volume Aqueous	% As^{76} Recovery	% P^{32} Recovery
.5	88 ± 1.8	90 ± 1.6
.3	57 ± 2.2	78 ± 2.0

2. Effect of acid concentration on extraction of arsenic^{76}. (See figure V.)

D. Calculations

1. Phosphorous-32

$$\text{uuc/ml } P^{32} = \frac{\text{uuc}^{*}\ \text{c/m x c/m at reference time}}{\text{Sample size (ml) x decay factor}}$$

2. Arsenic-76

$$\text{uuc/ml } As^{76} = \frac{\text{uuc}^{*}\ \text{c/m x net c/m x geometry factor x } \frac{3}{2}^{**}}{\text{Sample size x decay factor}}$$

---

*Factor based on the efficiency of the crystals used in the counting instruments.

**Factor based on mounting $\frac{2}{3}$ of the organic.

FIGURE I

DECAY OF PHOSPHOROUS
COMPLEX AFTER SEPARATION

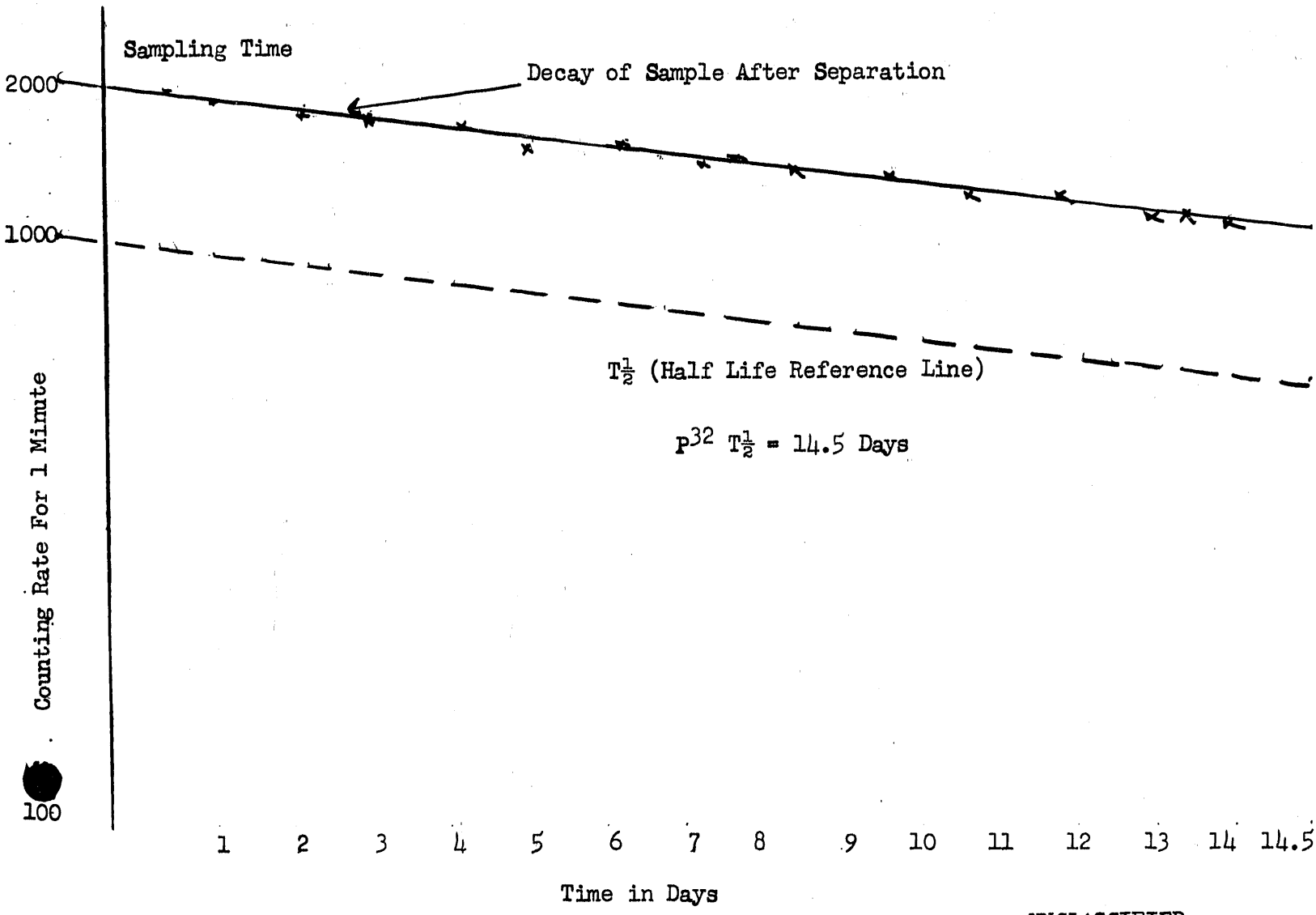
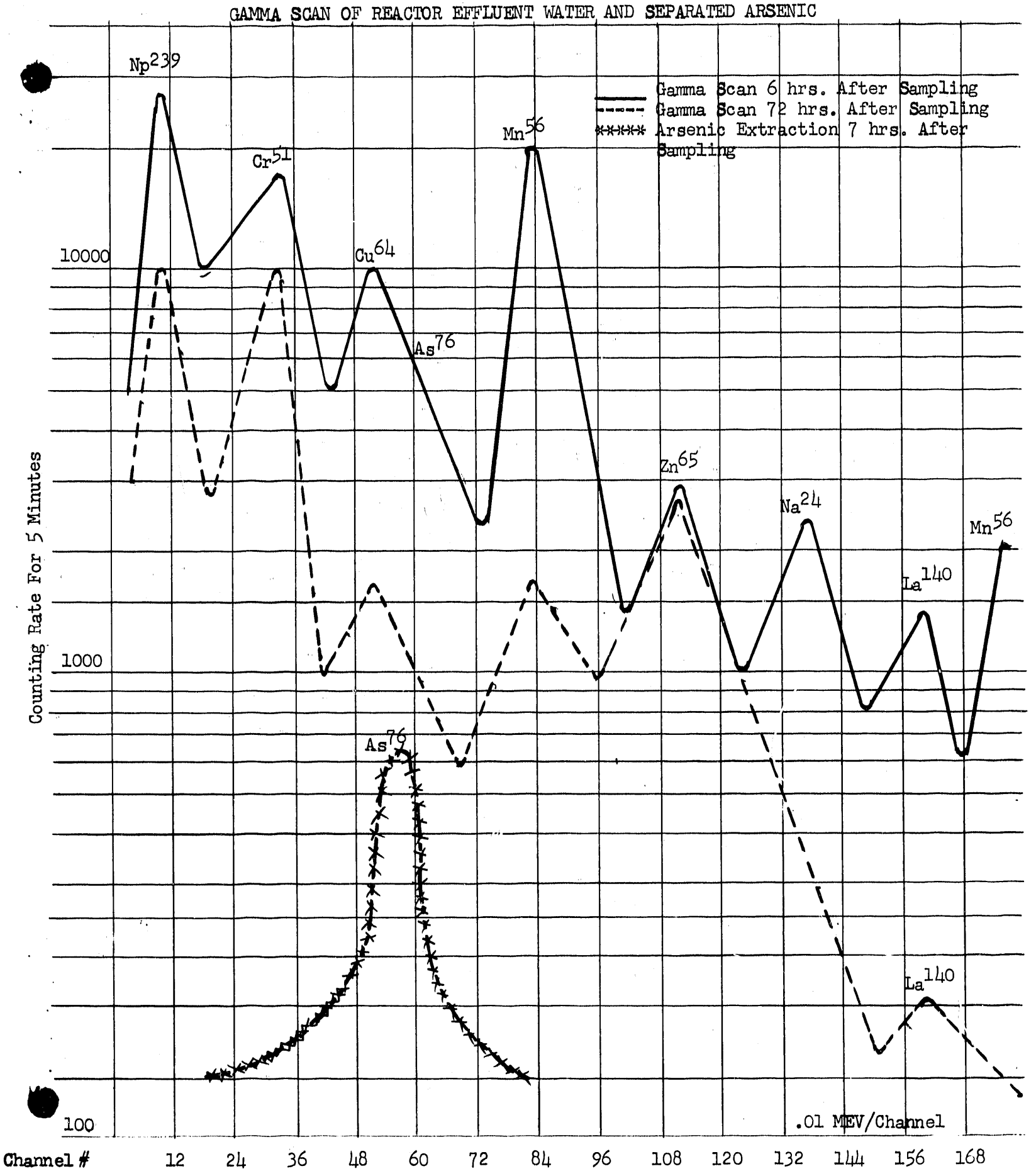


FIGURE II

GAMMA SCAN OF REACTOR EFFLUENT WATER AND SEPARATED ARSENIC



GAMMA SCAN OF REACTOR EFFLUENT
WATER AND SEPARATED ARSENIC
WITH FUEL ELEMENT RUPTURE

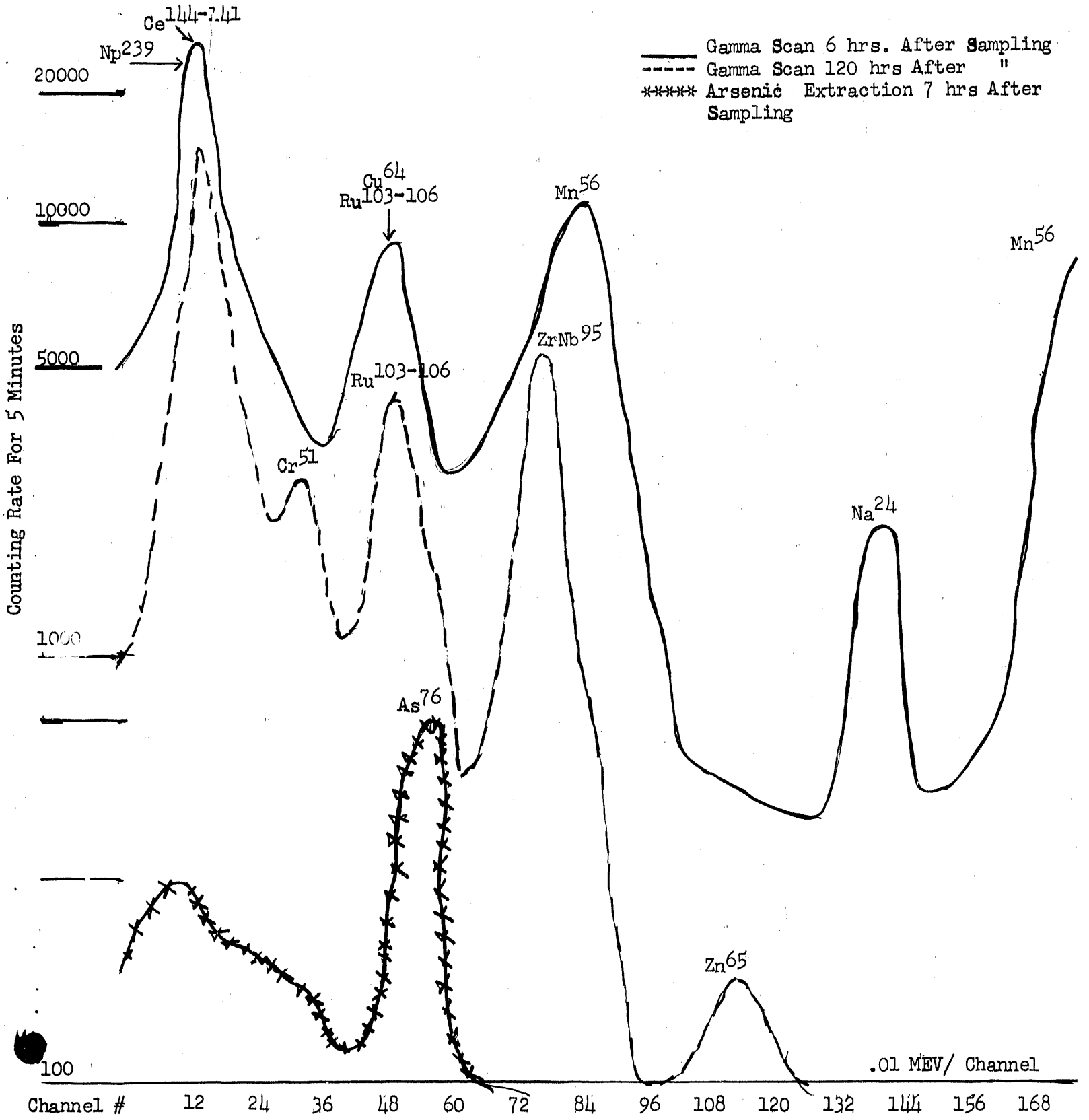


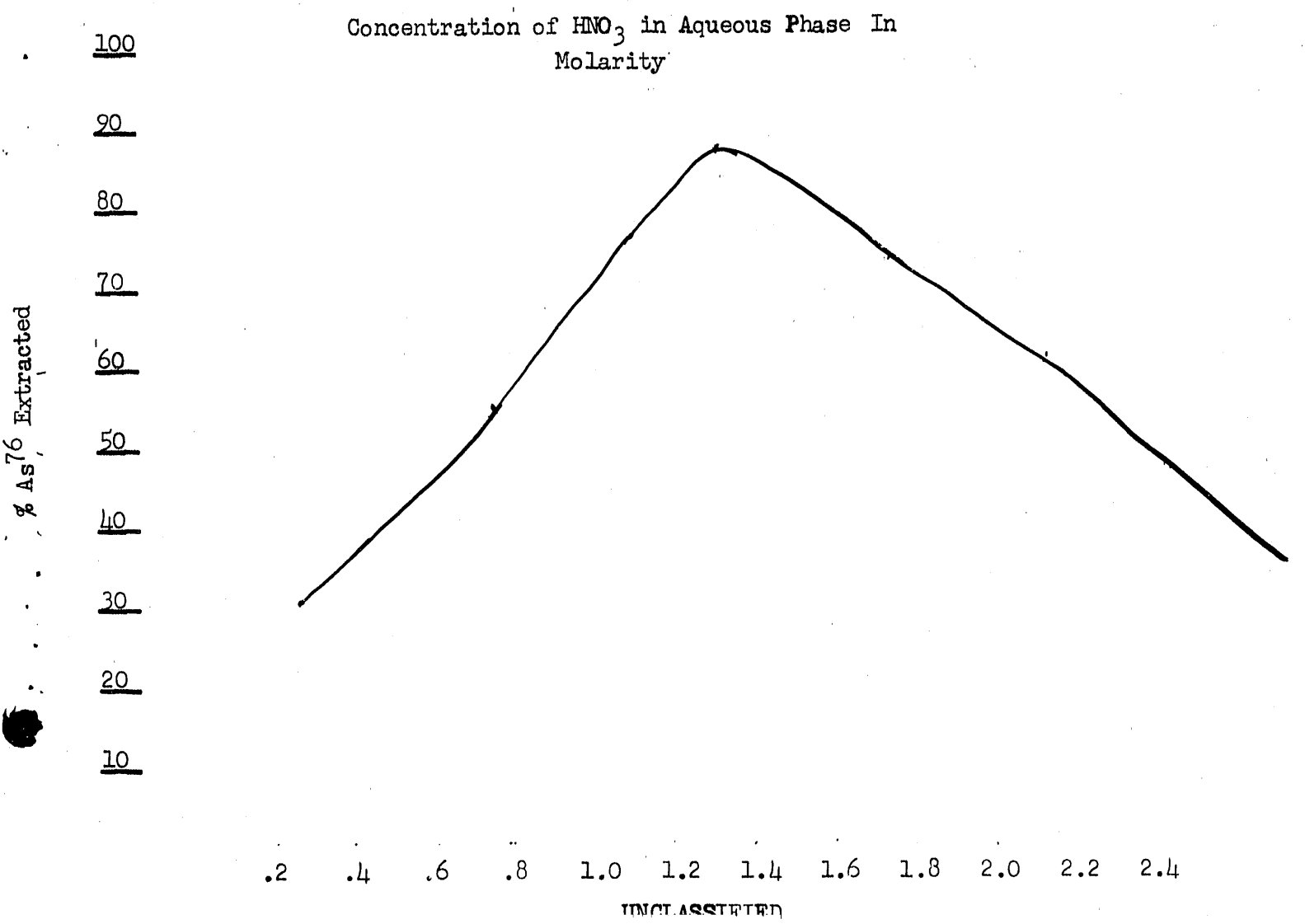
FIGURE IV

INTERFERENCES FROM OTHER RADIO-
ISOTOPES COMMON TO THE SAMPLE

Radioisotope	Gamma Energy	C/M Added	C/M Observed In Extraction	% Extracted
Cs^{137}	.66	2816	$<$ 5	$<$ .02
Ru^{103}	.49	3744	18	0.5
Sr^{85}	.50	337	$<$ 5	$<$ 1.5
Mn^{54}	.51	858	$<$ 5	$<$ 0.5
$Zr^{95}Nb^{95}$	.75	1236	$<$ 5	$<$ 0.4

FIGURE V

EFFECT OF CONCENTRATION OF ACID ON EXTRACTION OF ARSENIC^{76}



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