Title

A FILTRATION STUDY OF A SPARK GENERATED AEROSOL OF THE TRANSPLUTONIUM ELEMENTS

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University of California, Berkeley, California

June 7, 1951

ABSTRACT

The spectrographic analysis of transplutonium elements involves samples in the range of 1 to 50 micrograms with an activity level of approximately 10^8 c/m/sample. The finely divided radioactive aerosol produced by sparking represents a serious potential health hazard. A closed system type filter train has been developed to entrap this aerosol and to eliminate any danger to operating personnel. Incorporated into the filter system are check points that are utilized to establish a material balance within the train. The final check point, an evacuated cylinder, shows that 1 part in 10^{10} parts passes through the filtering train.

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INTRODUCTION

The spectrographic analysis of transplutonium elements involves samples of microgram amounts, the exact amount depending upon the quantity available. Curium samples are in the range of 1 to 2 micrograms -- this represents a specific activity of 3.9×10^9 c/m/microgram. Americium samples run as high as 50 micrograms where the specific activity is 3.2×10^6 c/m/microgram. The radioactive aerosol generated by sparking represents a serious potential health hazard to operating personnel.[1]

The following studies have arisen in an attempt to control this hazard and to determine the deposition of the active material within the filter train.

APPARATUS AND PROCEDURE

The equipment[2] (spark chamber, filter, etc.) was designed so as to form a closed system which is shown schematically in Fig. 1. The routine operation of this apparatus includes the following:

The active solution is dried on the flat end of a copper electrode and placed in the spark chamber where it is sparked[3] for 60 seconds. During the

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[1] "Handbook 42", National Bureau of Standards, p. 11, September, 1949.

[2] J. G. Conway, M. F. Moore, "The Spectrographic Analysis of Radioactive Materials", UCRL-1138, February 23, 1951.

[3] Applied Research Laboratory Spark Source Setting: 4 KVA
Inductance: 0.32 mh

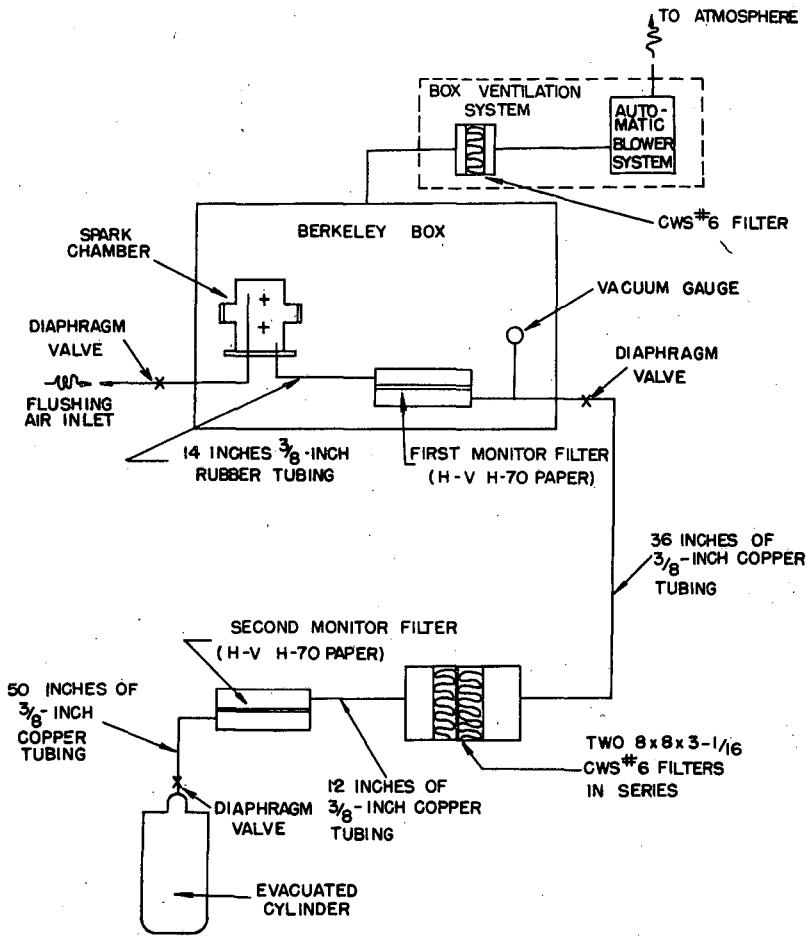
TO ATMOSPHERE
BOX VENTILATION SYSTEM
AUTO-MATIC BLOWER SYSTEM
CWS #6 FILTER
BERKELEY BOX
SPARK CHAMBER
VACUUM GAUGE
DIAPHRAGM VALVE
DIAPHRAGM VALVE
FLUSHING AIR INLET
14 INCHES 3/8-INCH RUBBER TUBING
FIRST MONITOR FILTER (H-V H-70 PAPER)
36 INCHES OF 3/8-INCH COPPER TUBING
SECOND MONITOR FILTER (H-V H-70 PAPER)
50 INCHES OF 3/8-INCH COPPER TUBING
TWO 8x8x3-1/16 CWS #6 FILTERS IN SERIES
12 INCHES OF 3/8-INCH COPPER TUBING
DIAPHRAGM VALVE
EVACUATED CYLINDER

FIG. 1

MU 2064

sparking the chamber is constantly flushed with air to prevent window fogging and to remove the active aerosol. The air is brought in near the roof of the chamber and withdrawn at the base.

From the base the air is drawn into the first monitor filter unit which is composed of two shells[4] enclosing a piece of 4 1/2 x 9 inch Hollingsworth and Vose H-70 paper. The line then passes out of the Berkeley Box to the main filter unit[5] which is composed of two 8 x 8 x 3 1/16 inch CWS No. 6 filters connected in series. The air passes into a second monitor filter unit, a duplicate of the first, and the system is terminated in an evacuated cylinder.

The entire filter train is operated under a slight negative pressure (less than 1 inch of mercury) and is tested for tightness by evacuating to approximately 28 inches of mercury. The philosophy is that, if the system is vacuum tight, the activity will not escape. Moreover, if operated under a slight negative pressure, any leaks that develop will be into the filter train and not into surrounding atmosphere.

EXPERIMENTAL

Since the apparatus exists primarily for analytical purposes, the experiments are not of a simple nature. In addition, the complexity of the experiment makes it difficult to perform all the tests on a single sample. During all tests, Filter Queen monitoring units using Hollingsworth and Vose H-70 paper monitored room air at the rate of 4 cfm and, for all runs, these papers showed zero c/m above counter background.

Experiment I

While the equipment was new several counting discs of one-inch diameter

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[4] UCRL Sketch No. HC-2850, HC-2950, HC-3450.

[5] UCRL Sketch No. HC-3250, HC-3350, HC-3450.

were placed inside the spark chamber and several samples of plutonium and americium were sparked.[6] The activity on all plates, except one, jammed the counter. This plate, equated against chamber area, indicated a total contamination of 0.21×10^8 c/m or approximately 7.4 percent of the activity sparked.

The activity collected on the first monitor filter was lost in chemical separation. The first CWS filter was digested and counted for americium. It showed 0.61×10^8 c/m or 18.7 percent of the total sparked. The second monitor filter showed zero c/m. The evacuated cylinder was not analyzed since it had been previously used on a less efficient filter train.

Experiment II

This experiment was run to supply the information not available in Experiment I.

A one-microgram sample of americium was sparked.[6] The first monitor filter was chemically processed[7] and showed 2.4×10^6 c/m collected or 73 percent of the americium sparked. The second monitor filter showed zero c/m. The evacuated cylinder, in this case a new glass carboy, showed[8] zero c/m.

Experiment III

Due to the high level of contamination on the walls of the spark chamber, it was felt some material was driven into the filter train by the spark blast.

Clean blank electrodes were sparked.[6] Recovery from the first monitor filter showed 0.43×10^6 c/m of americium. Using this as a correction fac-

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[6] Flow Rate through System -- 1.25 cfm.

[7] Chemical Procedure Appendix I.

[8] Chemical Procedure Appendix II.

tor for Experiment II we have, instead of 73 percent collection, only 60 percent collection of the amount sparked. The second monitor filter and evacuated cylinder showed zero c/m. The results of these three runs are tabulated below.

Table I

Spark Chamber	7.4 %
First Monitor Filter	60.0 %
CWS Filters	18.7 %
Second Monitor Filter	0 %
Evacuated Cylinder	0 %
	86.1 %*

* This figure is based on 95 percent chemical extraction yield from the filter paper.[7]

SUBSEQUENT EXPERIMENTS

It was felt that the amount of material deposited on the first monitor filter might be a function of the flow rate through the spark chamber. Sparked aerosols of varying size were filtered at different flow rates.[9] The first monitor filters were chemically extracted and the results plotted. (Fig. 2) An exact and rigorous interpretation of this curve is not possible because there has been no measurement of the particle sizes involved. Possible interpretation could include:

(a) More complete flushing of spark chamber,

(b) Favorable variation in flocculation scheme of particulate matter.

A two-microgram curium sample was sparked. This represents an activity of 7.8×10^9 c/m. In this run the glass collection carboy (evacuated cylinder) showed 0.6 c/m or 1 part in 1.3×10^{10} parts sparked. This is the only experimental run in which activity was found in the carboy. The second monitor filter showed zero c/m. This latter reading is not surprising

[9] Measured with Emil Greiner "Rotameter" Flowmeter No. G-9148.

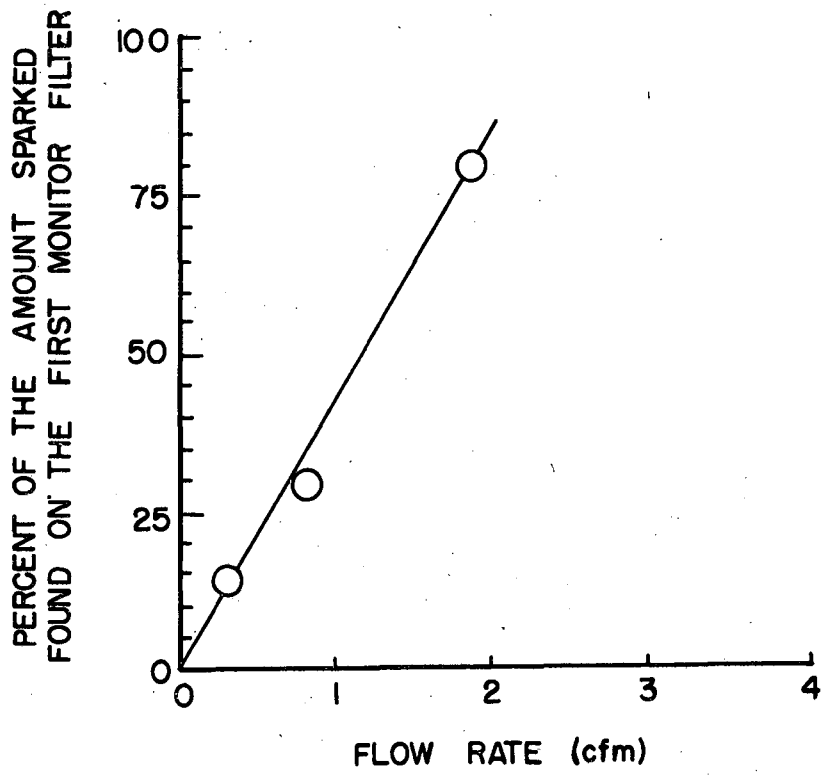
PERCENT OF THE AMOUNT SPARKED FOUND ON THE FIRST MONITOR FILTER
100
75
50
25
0
0
1
2
3
4
FLOW RATE (cfm)

FIG. 2

MU 2065

because of the difficulty of counting very low levels of activity on H-70 type paper.

CHEMICAL PROCEDURES - I

The filter paper to be recovered is folded to fit into a 200 ml beaker, approximately 25 ml of concentrated nitric acid is added and the wetted paper shredded. The beaker is placed on a hot plate and the temperature adjusted so that the slurry simmers. After 15 minutes of heating, the filter paper slurry is filtered and washed with nictric acid. The total volume of the filtrate is measured, and counting aliquots are taken. Aliquots are chosen to keep the counting rate within the range of the instruments. For highly active samples this may require dilutions. The extraction yield from the paper is in the order of 95 percent as determined by re-extraction. This procedure does not extract plutonium.

CHEMICAL PROCEDURES - II

A new, clean evacuated carboy was used as the collection tank. Before evacuation, 30 ml of concentrated nitric acid was placed in the carboy so that the atmosphere would be a saturated vapor. After collection the carboy, still under a slight vacuum, was alternately cooled and heated to condense out the activity with the nitric acid. Upon opening the carboy was flushed with clean air, the effluent air being passed through a Hollingsworth and Vose H-70 paper filter. This paper showed zero c/m in all cases. The nitric acid in the cylinder was poured into a beaker and this acid along with subsequent washes was slowly evaporated, the final amount being plated, flamed and counted.

This work was performed under the auspices of the Atomic Energy Commission.

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