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FRACTIONAL AIRBORNE RELEASE
OF STRONTIUM DURING THE COMBUSTION
OF 30 PERCENT NORMAL TRIBUTYL PHOSPHATE
IN A KEROSENE-TYPE DILUENT

Battelle

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FRACTIONAL AIRBORNE RELEASE OF STRONTIUM DURING THE COMBUSTION
OF 30 PERCENT NORMAL TRIBUTYL PHOSPHATE IN A KEROSENE-TYPE DILUENT

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This report is a research study performed as part of the program, "Fire Protection in Caves, Canyons, and Hot Cells," conducted by Atlantic Richfield Hanford Company for the Division of Operational Safety, United States Atomic Energy Commission.

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ABSTRACT

Engineering scale experiments were performed to determine the fractional airborne release of strontium during a fire involving a contaminated, flammable solvent. Earlier laboratory scale experiments using radioactive materials indicated that this release would be one percent or less. [1]

Twenty-five grams of strontium as strontium nitrate was added to 150 liters of 30% normal tributyl phosphate in a kerosine diluent, which was then burned in the ARHCO 224-T Fire Test Facility. Temperatures of 1000°C were recorded during the fire, the maximum scale of the instruments. There were 2 successful fires lasting 70 and 51 minutes, both burning to the point of self-extinguishment. 0.2% of the strontium was released in both burns. The bulk of the released strontium measured was collected in bubbler samplers installed upstream of the sprayed chamber air washer, indicating that the water sprayed in this chamber scavenged the strontium from the air stream.

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INTRODUCTION

As nuclear reactors provide an increasing portion of the nation's growing energy demands, the behavior of radioactive materials contained in fuel during various parts of the fuel cycle is of concern. An important segment of the cycle is the reprocessing step in which fissile fuel constituents are reclaimed through chemical separation steps. Liquid-liquid extraction using normal tributyl phosphate (TBP) in a kerosene diluent*, normal paraffin hydrocarbon (NPH) is commonly used in separating fissile and fertile materials from waste fission products generated in reactors.

In a typical Purex fuel reprocessing plant uranium and plutonium are decontaminated, separated, and purified by solvent extraction. In this process, the elements of valence IV are relatively extractable while the elements of valence III are relatively unextractable. Nitric acid is used as a salting agent. Primary separation of the uranium and plutonium from the radioactive waste occurs in the initial cycle of the process -- co-decontamination. Greater than 99.9% of the fission products are removed into the aqueous waste stream. The uranium, plutonium and remaining fission products are found in the organic product stream. This organic phase contains the highest concentration of total radioactive material found in an organic phase in the Purex process. Among the radioactive materials of interest in separation processes are ^{137}Cs , ^{131}I , $^{144}\text{Ce-Pr}$, and $^{95}\text{Zr-Nb}$.^[2] Many other fission products are present; those named are representative of chemically-similar groupings of the spectrum of the fission products present.

The organic solvent, 30 percent TBP in kerosene, is of limited flammability. TBP has a flash point of 295°F, Class I flammability, and kerosene has a flash point of 100°F, Class II flammability.^[3] There are some indications that the kerosene under process conditions of saturation with water is flammable over a limited range of temperatures.^[4] Low probability fire-initiating events may nevertheless occur and lead to combustible vapor formation which

* "Kerosene", and "NPH", are used interchangeably in this report for the TBP diluent. The specifications denoting its detail composition is given in Appendix A.

sustains burning over extended periods. Work earlier reported describes large scale fire characteristics. [5]

OBJECTIVE

The objective of this study was to measure the fractional airborne release of strontium during the burning of 30 percent n-TBP in kerosine.

SUMMARY AND CONCLUSIONS

Three engineering scale experiments were conducted to measure the fractional airborne release of strontium during the combustion of 30% n-TBP in NPH solution simulating the organic product from the Purex decontamination cycle. Because short sampling times and low recoveries were anticipated, rather high concentration of strontium in the organic solvent was used. An almost saturated solution of strontium nitrate in 100 ml of water was mixed with the TBP-NPH. 150 liters of this solution were burned in a sweep of air. Samples were taken at two places in exhaust duct, upstream and downstream of the sprayed air washer.

The samples taken ahead of the washer were at a high temperature, e.g., 800°C and so the sample was cooled by using a bubbler upstream of the filter sampler. The strontium residue in the tubing, bubbler and on filter paper samples was measured.

Thus, non-radioactive strontium was used as representative of radioactive materials of limited volatility found in contaminated process solvents. It will act in a manner similar to the radioactive isotope.

ANALYTICAL

Atomic absorption spectrophotometry and X-ray fluorescence provide sensitive analytical methods for strontium and were employed in the study.

X-ray fluorescence analysis provides definite quantitative measurement of certain elements, including strontium. The aerosol sample is collected on a membrane filter, the filter is then mounted and subjected to X-ray excitation and the fluorescence measured. The method is rapid and non-destructive, and as used in this experiment gave a lower detection limit of 10^{-9} g/cm².

Atomic absorption spectrophotometry is a quantitative technique for the determination of elements in liquid and solid samples. The sample is heated to a high temperature, breaking the bonds between the molecules, enabling individual atoms to float freely in the sample area. The atoms can absorb ultraviolet or visible radiation. The wavelength bands which each specific element can absorb are very narrow and different for each element. The lower detection limit for strontium in this study by atomic absorption is 0.1 ppm, measured in the solution or material for analysis.

EXPERIMENTAL

As part of a larger study of "Fires in Caves, Canyons and Hot-Cells"^[5,6,7,8,9] three engineering scale experiments were performed to measure the fractional airborne release of stable isotopes of strontium.

Bench scale experiments had been performed to measure fractional release of radionuclides during combustion of process solvent. Radionuclides were used to represent various significant classes of radioactive materials normally found in process solvents. Uranium at nominally anticipated concentrations was used to represent extractable transuranics. Most abundant radionuclides were used at tracer levels to represent non-extractable elements of various degrees of volatility. Thus ^{131}I was used to represent volatiles, ^{137}Cs represented the semi-volatiles and ^{95}Zr and ^{144}Ce represented non-volatiles.^[1]

Since a facility in which realistic simulation of a full-scale cell fire could be performed was readily available (the ARHCO 224-T Fire Test Facility) corroboration of the bench-scale fractional release values was desirable. The facility is not suitable for use of radioactive materials and thus a stable simulant was necessary. A combination of limited solubility and analytical sensitivity precluded the use of many of the stable isotopes of the fission radionuclides. Strontium, a material not as abundant in process solvents as many others, but with a radioactive isotope, ^{90}Sr , which is radiologically significant, was found to have the requisite solubility in process solvent and could be measured at anticipated levels using X-ray fluorescence and atomic absorption.

Facility

The ARHCO Fire Test Facility is diagrammed in Figure 1 and consists of:

- a. A 12' x 12' cell of variable height of insulating board housed in a sheet steel silo.

FIGURE 1

DIAGRAM OF FIRE TEST FACILITY.

- b. A fire extinguishment test system.
- c. An exhaust system composed of a 1.5' x 2' duct following by a water spray cooling section, a filter test section exhaust fan, velocity sensing element, and short stack.

The test facility has instrumentation to measure temperature, pressure, flow, smoke and oxygen content at various points in the system. The nominal flow through the system is 2000 cfm resulting in nominal velocities of approximately 14 fpm in the cell and 670 fpm in the exhaust duct. For these experiments air was drawn into the cell from the top and exhausted through the horizontal duct at the exhaust point about four feet above the floor.

Experimental Procedure

Three experiments were performed using the following procedure. The test solvent was arranged in ten stainless steel trays 17" x 23" x 3" deep. 150 liters of 30% TBP-NPH containing 25 g of strontium was distributed evenly to the trays. They were positioned to the side of the hot cell nearest the air exit to aid the burning. Cement blocks held them 16" above the hot cell floor and 8" above the surface of water placed on the floor to provide protection for the heat.

NPH was pumped into the cell on the water surface to aid ignition. Gasoline soaked rags were set on fire with a torch and the burning bundle put on a cement block in the silo near the trays to start the fire. Airflow (2000 cfm) was begun, pulling the flames down the duct.

Description of Burning

Burn #1

This burn was unsuccessful, reaching an air temperature of only 150°C, with three trays burning. The sampling periods were too short and yielded no meaningful results. However, this experiment helped to redesign the experiment for two successful burns.

Burn #2

The fire was started at 1:25 p.m., reaching temperatures of 100°-200°C. At 1:34 the fan stopped for three minutes (samplers were turned off during this time). Air flow began again at 1:37 and the sampling was begun again.

The temperature at that time in the duct was 200°C and at 1:55 the temperature had reached 1000°C where the instruments pegged for five minutes. The temperature then gradually subsided with the fire burning itself out at 2:35.

Burn #3

The fire was ignited at 1:26 p.m. with the temperature rising quickly to 1000°C, five minutes after the run began, where the instruments pegged out. The air flow was cut down at 13.5 minutes into the run, but the fire continued to burn vigorously. The high temperature lasted for 20 minutes, then had a slight drop to 900°C, then returned to 1000°C at 2:00. Temperatures began to fall at 2:10 and the fire extinguished itself at 2:17.

Smoke Samples

Samples were taken at points upstream and downstream of the sprayed chamber air washer. The samplers are shown in Figure 2.

Upstream (A) Samplers

A 1/2 inch diameter stainless steel sample probe was extended to the middle of the horizontal exhaust tunnel. A long-radius bend near the end permitted the probe to be faced directly into the air stream. Air was drawn from the hot stream at 2 cfm, measured at ambient temperature and pressure, and bubbled through 4-liter suction flasks filled with distilled water acidified with HNO₃. Precleaned stainless steel wool was inserted into the tubing and around the bubbler end of tubing to filter soot from the airstream. An in-line filter* holder containing a 0.65 µm pore size Millipore filter** was located downstream of the bubbler. The airstream was heated mildly with a propane torch just ahead of the filter holder to avoid moisture condensation.

Downstream (B) Samplers

These samplers were similar to the upstream samplers but without the bubbler. This airstream was also heated to avoid moisture condensation.

* Model #710 in-line filter holder, Environmental Research Corporation, 3725 N. Dunlap St., St. Paul, Minnesota 55112

** Millipore filter, type DA, 0.65 µm, Millipore Corp., Bedford, Mass. 01730.

FIGURE 2
SAMPLERS USED IN MEASURING FRACTIONAL AIRBORNE RELEASE

Four samples were taken at each location during the run. The samples were spaced at intervals to cover all phases of the experiment. Sampling time ranged from 30 seconds to 11 minutes. The stainless steel tubing was rinsed with acidified distilled water which was boiled and concentrated. For the A (upstream) samples, the washings were combined with the bubbler solutions and concentrated (except for burn 2, 2A where the 2 samples were analyzed separately to see if strontium deposition on the sampling line varied from the bubbler concentration). The solutions were analyzed by atomic absorption and the filter papers measured directly by X-ray fluorescence techniques.

Residues

The fires burned to a point of self-extinguishment so the residue from the burns was available for analysis. There were two residues:

1. Seven trays in the hot cell near the duct exit burned to a point of nominal dryness.
2. The four trays near the central core of the hot cell burned only to where there was a thick syrupy liquid left.

The dry residue was weighed, an aliquot of this total amount taken for analysis, the liquid residue was measured and an aliquot taken for analysis. Recovery was 49 and 47 percent of strontium initially present.

The residue was hygroscopic as well as non-uniform, so consistent residue analyses could not be expected.

RESULTS AND DISCUSSION

The strontium analysis of the various sampler samples is shown in Table I. The fractional release of strontium during the run did not vary greatly from that indicated by earlier laboratory work. In Burn #2, 0.22% of the strontium was released; Burn #3, 0.19%. These results come exclusively from the A samplers, upstream of sprayed washer, and there principally from the bubblers, indicating the solubility of the strontium in water. The filters showed strontium, but in most cases not enough to quantify.

The samples taken downstream of the sprayed air washer indicated the presence of strontium but were significantly lower than the bubbler samplers, leading to the conclusion that the washer scavenges contaminants from the system.

There was no time period during the burns during which a significantly greater than average release rate of strontium occurred.

The scope and funding level of this study did not permit a parametric assessment of the release of the many fission products which could become airborne in solvent fires. Besides the volatility properties of the elements and compounds which may be involved, one must also acknowledge a wide variation in rate of burning, ventilation rates, and air sweep patterns which may affect the release fraction. The burning during these two experiments is regarded as about as vigorous as might be experienced in a hot cell fire, hence the release of a few tenths of one percent of strontium is believed to represent more nearly a maximum than a minimum value from a burning pool of TBP-kerosine.

We recommend a series of experiments be conducted to define a range of release values as a function of element and burning conditions.

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FIGURE 3

RESIDUAL MATERIAL FROM BURN #2

FIGURE 4

BURN #3 - SIX MINUTES AFTER IGNITION OF FIRE

FIGURE 5

BURN #3 - 32 MINUTES AFTER IGNITION OF FIRE

TABLE I
RESULTS OF TWO BURN EXPERIMENTS

SAMPLE NUMBER		SAMPLING TIME (MIN)		SAMPLING DURATION (MIN)	Sr CONC IN [†] WASHINGS AND BUBBLER, $\mu\text{g/cc}$	Sr IN BUBBLER AND WASHINGS, μg	Sr ON FILTER, ng/cm^2	AREA OF FILTER, cm^2	Sr ON FILTER, μg	TOTAL AIR SAMPLED ft^3	TOTAL Sr ^{††} IN AIR PASSING IN DUCT DURING SAMPLING TIME, μg	FRACTION OF Sr ^{†††} PER MINUTE DURING SAMPLING TIME	DURATION OF THIS RELEASE RATE, MIN	FRACTION OF Sr RELEASED TO AIR DURING THIS PERIOD	ACCUMULATIVE FRACTION RELEASED												
		ON	OFF																								
BURN #2,	1A	5.2	7.2	6.5	0.3 ± 0.05	3	15 ± 3	44.1	0.66	13	3700	3x10 ⁻⁵	14.5	4x10 ⁻⁴	4x10 ⁻⁴												
UPSTREAM OF SPRAY WASHER SECTION	2A	20	31													11	0.5 ± 0.05	5	20 ± 13	44.1	0.88	22	5900	3x10 ⁻⁵	16.5	5x10 ⁻⁴	9x10 ⁻⁴
	3A	35	45													10	0.6 ± 0.05	6	<13	44.1	<0.57	20	6000	3x10 ⁻⁵	14	4x10 ⁻⁴	1.3x10 ⁻³
	4A*	50	57													7	0.4 ± 0.05	4	<13	44.1	<0.57	14	4000	3x10 ⁻⁵	25	8x10 ⁻⁴	2.2x10 ⁻³
DOWNSTREAM OF SPRAY WASHER	1B	5.25	7.25	5.7	<0.04	<0.4	<5	51.6	<0.26	11	<400	<3x10 ⁻⁶	15	<5x10 ⁻⁵	-----												
		11.3	15.0																								
	2B	20	28													8	<0.04	<0.4	<5	51.6	<0.26	16	<400	<3x10 ⁻⁶	13	<4x10 ⁻⁵	-----
	3B	35	40.5													5.5	<0.04	<0.4	<5	51.6	<0.26	11	<400	3x10 ⁻⁶	12.5		
	4B*	45	49	4	<0.04	<0.4	17.7 ± 7	51.6	0.91	8	900	0.9x10 ⁻⁶	29.5	6x10 ⁻⁵	-----												
BURN #3, UP STREAM OF SPRAY WASHER SECTION	1A	5	7.5	2.5	0.18 ± 0.05	1.8	<14	44.1	<0.62	5.0	1800	6x10 ⁻⁵	7.5	5x10 ⁻⁴	5x10 ⁻⁴												
	2A**	14	15.5													1.5	0.18 ± 0.05	1.8	<15	44.1	<0.66	3.0	1800	9x10 ⁻⁶	8.0	7x10 ⁻⁴	1.2x10 ⁻³
	3A	36	43.5													7.5	0.09 ± 0.05	0.9	<12	44.1	<0.53	15.0	900	9x10 ⁻⁶	28	3x10 ⁻²	1.5x10 ⁻³
	4A***	51	56													5.0	0.18 ± 0.05	1.8	<11	44.1	<0.47	10.0	1800	3x10 ⁻⁵	12.5	4x10 ⁻⁴	1.9x10 ⁻³
DOWNSTREAM OF SPRAY WASHER	1B	5	5.5	0.5	<0.05	<0.5	<11	51.6	<0.57	1	<107	<8x10 ⁻⁶	5.5	<4x10 ⁻⁵	-----												
	2B**	14	16.5													2.5	<0.05	<0.5	<12	51.6	<0.62	5	<112	<1.8x10 ⁻⁵	11.0	<2x10 ⁻⁴	-----
	3B	36	39.5													3.5	<0.05	<0.5	26 ± 11	51.6	1.3	7	1300	3x10 ⁻⁵	23.0	7x10 ⁻⁴	7x10 ⁻⁴
	4B***	51	56													5	<0.05	<0.5	<12	51.6	<0.62	10	<112	<9x10 ⁻⁵	16.5	<1.5x10 ⁻³	

* FIRE BURNED 71 MINUTES

** THE AIRFLOW IN DUCT WAS SLOWED AT THIS POINT, BUT FLOW READING INSTRUMENTATION DID NOT OPERATE

*** SAMPLING CONTINUED AFTER FIRE WENT OUT AT 51 MINUTES

† SCRUBBER SOLUTION WAS CONCENTRATED TO 10cc VOLUME

†† ALIQUOT SAMPLED WAS $\frac{2 \text{ CFM}}{2000 \text{ CFM}} = 0.001$

††† BASED ON Sr DISSOLVED IN TBP-NPH AT BEGINNING OF THE RUN

TABLE IIANALYSIS OF ORGANIC SOLVENT

<u>Burn</u>	<u>% TBP</u>	<u>% NPH</u>
1	40	60
2	23.3	26.5
3	32.3	67.7

TABLE IIIMETEOROLOGY

<u>Burn</u>	<u>Date</u>	<u>Temperature °F</u>	<u>Relative Humidity</u>	<u>Station Pressure</u>	<u>Wind MPH</u>
1	2-7-74	50.4	45	29.75	SE5
2	3-14-74	45	46	29.33	NW9
3	3-25-74	56	59	29.76	WSW1

APPENDIX AMATERIAL SPECIFICATIONS

Chemical: Normal Paraffin Hydrocarbon (NPH)
 CPD Material No.: 669

<u>Purchase Specifications</u>	<u>Value Wt.%</u>	<u>Test Requirements</u>	
		<u>Method</u>	<u>Frequency</u>
a. N-paraffin hydrocarbon	Min. 98 vol.%	Zy-28	ES
b. C ₁₀ to C ₁₄ components	Min. 99 vol.%	Zy-28	ES
c. Aromatics as 1,2,3,4-tetrahydronaphthalene	Max. 0.2 Wt.%	Zz-9	ORPE
d. Iodine number (olefins as wt.% 1-tetradecene)	Max. 0.1 wt.%	Zx-21	ORPE
e. Alcohol	Max. 0.05 wt.%	Zx-22	ORPE
f. Sp. Gr. 60/60 F	Max. 0.76	Zy-10A	ES
g. Viscosity @ 25°C	Max. 1.8 centipoises	Zy-9	ES
h. Flash Point	Min. 70°C	Zy-6	ES
i. Interfacial tension between hydrocarbon and 0.1M NaOH	Min. 12 Dynes/cm	Zy-22	ORPE
j. Solids	None Visible	Visual	ES
k. Color	Colorless	Visual	ES

NOTE: Vendors whose products meet the above specifications may submit a one-gallon sample for laboratory evaluation of compatibility with plant processing conditions.

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