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**FINES ANALYSIS OF PLUTONIUM DIOXIDE
MICROSPHERES AFTER IMPACT**

MASTER

P. H. Bonnell

AEC Research and Development REPORT

MONSANTO RESEARCH CORPORATION

A SUBSIDIARY OF MONSANTO COMPANY

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MIAMISBURG, OHIO

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MONSANTO RESEARCH CORPORATION

A Subsidiary of Monsanto Company

MOUND LABORATORY

Miamisburg, Ohio

operated for

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ABSTRACT

Seven capsules containing sized $^{238}\text{PuO}_2$ microspheres were impacted into concrete, granite, and sand targets at velocities from 102 to 456 ft/sec (31 to 139 m/sec). The damage to the microspheres and percentage of fines produced were determined. The percentage of fines (particles 0-44 μm in diameter) increased with increasing velocity to a maximum percentage between 50-55%.

INTRODUCTION

With $^{238}\text{PuO}_2$ microspheres being used as the fuel in heat sources designed to generate electricity for more space missions it is important that the impact characteristics of the fuel be known. The release of microscopic radioactive particles through melting of the capsule (as could occur during launch pad explosion and/or fire) or fracture of the capsule on impact with the earth could create a hazard. Impact could occur after either suborbital or orbital abort of the vehicle. A cooperative program to study the formation of fines (particles less than $44\text{ }\mu\text{m}$) during the impact of microspheres was conducted by Mound Laboratory and Sandia Corporation. Mound Laboratory loaded the capsules and characterized the fuel both before loading and after impact. Sandia impacted the capsules at their test range.

Results of previous work at 248 and 377 ft/sec (76 and 115 m/sec) were reported.¹ In studies covered by this report, all capsules were impacted in an end-on mode. Various targets were used in an effort to study the amount of fines generated by a number of potential earth targets.

PROCEDURE

All work was completed according to the following schedule:

A. Before the capsules were loaded:

1. The microspheres were ultrasonically washed in alcohol.
2. The microspheres were screened and each size range weighed. This gave the percentage in the various size ranges before impact.
3. The microspheres were thoroughly remixed and loaded into the impact capsule (Figure 1).

B. After Impact:

1. The capsules were opened and the contents separated into two fractions:
(a) $44 - >177\text{ }\mu\text{m}$

FIGURE 1 - Drawing of impact capsule.

(b) <44 μm

2. The fraction greater than 44 μm was screened into the same size ranges as before loading. Each range was weighed and a sample removed for photomicrographic study. The six size ranges were:

177->200 μm	105-125 μm
149-177 μm	77-105 μm
125-149 μm	44-77 μm

3. The fraction less than 44 μm was placed in ethyl alcohol and screened into the following ranges:

20-44 μm
10-20 μm
<10 μm

All screen sizes are nominal values.

4. Samples from the two ranges greater than 10 μm were photomicrographed and the particles sized.
5. Total weight of these samples was determined by radiochemical analysis.
6. Samples from the <10 μm range were mounted on slides for particle size determination by Tracerlab.* The weight of the remainder was determined by radiochemical analysis.

PREPARATION OF CAPSULES

All microspheres were washed by the following method:

The spheres were placed in a Petri dish and ~100 ml of ethyl alcohol added. After a few minutes of ultrasonic cleaning, the liquid was decanted. This process was repeated three or four times. After each wash, a drop of liquid was placed on a filter paper and dried. The filter paper was then counted with an Eberline PAC-1SA portable monitor. Washing was stopped when the counting rates stabilized. Then 5 ml of ethyl alcohol was added and the dish gently rocked back and forth by hand. This liquid was decanted and added to the previous supernatant liquid.

*A Division of Laboratory for Electronics, Inc., 2030 Wright Avenue, Richmond, California.

The spheres were then allowed to dry by self-heating, weighed, and placed in an impact capsule. The capsule cap was electron beam welded. The capsule was helium leak tested, radiographed to determine weld integrity and fuel level, and calorimetered. The capsules were then shipped to Sandia Corporation, Albuquerque, New Mexico, for impact into various targets. All capsules were impacted end-on, long axis perpendicular to face of target.

IMPACTED FUEL EXAMINATION

Method When an impact capsule is received for particle size analysis, the impact velocity is reviewed to determine whether the capsule will be opened in a fumehood or in a glovebox. From experience it is known that capsules impacted at less than 120 ft/sec (37 m/sec) can be opened in a fumehood with normal precautions. Capsules impacted above this velocity are opened in a glovebox.

A Con-O-Saw is used to cut off the welded end-cap, and the screw cap is removed. If the material will pour out, it is poured into a 44- μ m screen which is sitting in a dish of ethyl alcohol. This is placed in an ultrasonic bath and the fines separated from the large size material. The alcohol containing the fines is placed in a plastic bottle and the large material washed again. This step is repeated until tests show there is no increase in the activity in the alcohol. Fresh alcohol is used each time. The large material is then dry screened into each of the six size ranges.

The alcohol containing the fines is screened through 10 and 20 μ m screens. This is done wet to prevent any material from becoming airborne. A sample containing particles from 0-20 μ m is removed and sent to Tracerlab for size distribution analysis.² A small sample is also taken from the 10-20 μ m and 20-44 μ m ranges for size distribution at Mound. The alcohol containing a given size range is evaporated to ~50 ml. The alcohol is placed in beakers, the beaker covered with a watch glass, and the solution taken to dryness. The particulate matter is then dissolved in an HNO_3 -HF mixture. The HF is evaporated and sufficient 2M or 8M HNO_3 is added to give 50 ml of solution. For capsules 12 and 16 a further dilution was made.

Fuel Loss Capsules 12 and 16 were opened inside a glovebox because the fuel was compacted and would not pour out. After the welded end cap was cut off capsule 16, a second cut was made through the impacted end. During this operation the saw passed through the compacted fuel. This fuel was lost because the saw ejected it into the glovebox. The remaining fuel was pushed out or picked out and some loss occurred at this point. The impacted end of capsule 12 was not cut. The wooden handle of a Q-tip was used to pick the material out of the cavity. This material was placed in a plastic bottle and capped to prevent any fines from becoming airborne. This process was repeated until all the material had been removed. The fuel cavity in the capsule was then swabbed with alcohol and a Q-tip.

In these two capsules the fuel came out, primarily, as compacted chunks. These chunks consisted of unbroken spheres, large broken particles, and fines. They were placed in a 44- μ m screen in alcohol and ultrasonically agitated to separate them. With this type of agitation we felt that no additional fines would be created and no fines would become airborne. The large amount of fines produced during impact clogged the screens making separation difficult. Some loss occurred during this step. Some additional material was lost when it clung to the sides of the plastic collection bottles. The fines were difficult to dissolve. These particles are extremely light and had a tendency to climb the sides of the beaker, and all of them could not be washed down. This was another source of loss. The fuel above 44 μ m from capsule 12 was weighed before it was separated into the six size ranges. It was then screened in a normal manner and the material in each size range was then weighed. Three percent of the total fuel in the capsule was lost. The lost material was trapped in the openings in the screens. This is a common occurrence when any material is screened and has occurred with every capsule, but measured only once. If it is assumed that a 3% loss in screening is common to all capsules, then 98.7 to 99.5% of all material can be accounted for in capsules impacted at less than 120 ft/sec (37 m/sec).

Only under more carefully controlled conditions could it be possible to determine at what step material was lost and in what quantities. It is doubtful whether any true quantitative answers could be found as to the exact particle size lost. However, qualitative answers as to size ranges and amounts could be found. By relating these qualitative answers to particle size distribution from samples a good approximation could be made.

Very little material will be lost from low impact shots because of the small amount of fines present. However, an appreciable amount of material would be lost from high impact shots. Procedures used for capsule 15 (Appendix) indicate that approximately 2.5% of the material is not weighed or measured analytically.

RESULTS AND DISCUSSION

The impact velocities, targets, and percentage of fines in the three size ranges are summarized in Table 1. When capsule 12 was impacted the graphite shattered and the capsule rebounded with enough velocity to break the Lucite catchbox. Capsule 15 stuck in the graphite and chipped some of the concrete at the point of impact. This capsule as well as all others, except 11, rebounded only a few inches. Capsule 11 penetrated the sand target for a distance of 33 in. (84 cm). Capsule 8 was treated exactly the same as all others except it was not impacted. This capsule was used to determine whether fines were produced in handling or storage.

Examples of the amount of compaction found from measurements of x-ray pictures of typical capsules are given in Table 2. The density of the fuel was calculated from these measurements and is also given in Table 2.

Table 1

IMPACT VELOCITIES, TARGETS, AND PERCENTAGE OF FINES

Cap- sule	Velocity		Target	Fuel Age at Impact (mo)	Percent Fines in Size Range		
	(ft/sec)	(m/sec)			20-44 μm	10-20 μm	<10 μm
3	104.3	32	granite	24	2.5	0.35	0.15
4	116	35	concrete	5	4.6	0.72	0.007
5	102.4	31	granite	24	3.1	0.5	0.15
8	control		not impacted	5	0	0	0
11	102.7	31	sand	5	0	0	0
12	442	135	granite ^a	24	34.0 ^c	6.5 ^c	6.4 ^c
15	456	139	concrete ^b	15	21.41	15.6	12.72
16	317	96	granite	10	24.9 ^d	9.8 ^d	4.8 ^d

^a A 3/4 in. (1.9 cm) sheet of Hitco graphite taped to face of target, fibers in graphite were perpendicular to face of target.

^b A 3/4 in. (1.9 cm) sheet of Hitco graphite, with fibers running parallel to surface, taped over face of target.

^c Some of this material was held up on the screens and equipment used and some became airborne. By using the results from capsule 15 as criteria 1.7% should be added to the 20-44 μm range and 0.3% to each of the other two ranges.

^d Some of the fuel in this capsule was dispersed into the glovebox. Therefore these percentages are only indicative of the relative proportions of fines in each size range.

Table 2

MEASURED FUEL VOID AND CALCULATED FUEL DENSITY
BEFORE AND AFTER IMPACTION

Capsule	Fuel Void				Calculated Fuel Density ^a (g/cm ³)	
	Before (in.)	Impact (cm)	After (in.)	Impact (cm)	Before Impact	After Impact
3	0.275	0.699	0.300	1.065	6.6	6.6
16	0.565	1.434	1.650	3.191	6.6	9.3

^a Density of production spheres - 9.8-10.4 g/cm³.

Figure 2 is a plot of total fines produced as a function of the impact velocity. All targets used for capsules in this graph were granite. The target for impacts at 442 and 456 ft/sec (135 and 139 m/sec) had a 3/4 in. (1.9 cm) thick piece of Hitco graphite over the impact face.

FIGURE 2 - Total fines produced as a function of the impact velocity. Results at 248 and 377 ft/sec (76 and 115 m/sec) are from work done by Dr. Neal Goldenberg.

It is doubtful that there would ever be more than 50-55% total fines produced. During impact the fines produced fill the void space between microspheres and absorb the shock waves and cushion the unbroken spheres. This is evidenced by the large reduction in fuel length and the high fuel density in high velocity impacts. There is also visual evidence when the fuel is removed from the capsule; some of the fuel comes out as compacted masses and then falls apart in the alcohol with the release of both microspheres and fines.

Since no capsules were impacted at velocities between 116 ft/sec (35.3 m/sec) and 317 ft/sec (96.4 m/sec) during this series of tests no impact densities can be calculated. Figures 3 and 4 are typical of particles, in the size ranges shown, regardless of impact velocity. These pictures clearly show that fines may be spheres that have broken in half or have completely shattered into many small fragments. The high density and large masses of particles that have been observed, are a good indication that little more compaction could occur regardless of impact velocity.

FIGURE 3 - Fines produced during impact in 20-44 μm range. 100X

FIGURE 4 - Fines produced during impact in 10-20 μm range. 100X

Figure 5 is a cumulative plot of the two capsules impacted at the extreme ends of the velocity range. The curve for capsule 5 corresponds well with the curves reported previously.¹ The curve from a capsule impacted between 200-250 ft/sec (61-76 m/sec) would fall between these curves and give 20-30% total fines.

FIGURE 5 - Cumulative particle size distribution of all size ranges.

CONCLUSIONS

The higher the impact velocity the higher the total percentage of fines. However, the shape of fines produced are the same regardless of impact velocity within a given size range. There appears to be a significant difference in total fines produced by a difference of only 14 ft/sec (4.2 m/sec) at the low end of the range but with only one or two capsules impacted at any one velocity nothing really definite can be concluded. Any in-depth study would require much more work than has been done. A method is now available for complete recovery of all material and a thorough analysis.

No known future work is being planned.

ACKNOWLEDGMENT

Grateful acknowledgment is made to R. E. Hermetz and R. L. Wise for their work in opening the capsules and sizing the fuel and to W. H. Johnson for his photography.

REFERENCES

1. P. H. Bonnell, Particle Sizing of Plutonium Dioxide Microspheres After Impact, MLM-1499 (June 21, 1968).
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APPENDIX

ANALYSIS OF FINES FROM IMPACT CAPSULE #15

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Capsule #15 was impacted by Sandia Corporation at 456.1 ft/sec (139 m/sec) into a concrete target with a 3/4 in. (1.92 cm) thick piece of Hitco graphite taped over the target area; the fibers in the graphite ran parallel to the target face. Upon impact the capsule stuck in the graphite and knocked a shallow area out of the target.

The capsule was opened by cutting off the electron beam welded end cap using a new Con-O-Saw in an uncontaminated glovebox. Two air samplers were set up inside the glovebox and a 4-hr background sample was taken before opening the capsule and a 24-hr sample was taken after opening the capsule. The first cut was through solid metal and the second cut just touched the top of the screw cap. The cotton tip of a Q-tip was used to wipe around the area between the screw cap and the capsule wall. This was bagged out and counted on a PAC-1SA hand monitor. The Q-tip read 1500 counts/min. The capsule was bagged out of the glovebox and transferred to a specially built plastic enclosure inside a fumehood (Figure 1).

FIGURE 1 - Plastic enclosure used to contain any airborne material.

A specially made tool was used to remove the screw plug under ethyl alcohol. A pair of forceps were bent to hold the capsule while the fuel was removed. Some fuel poured out into the beaker of alcohol when the screw plug was removed. The remainder was removed by working the wooden portion of a Q-tip into the fuel, with alcohol in the cavity, placing the end of the capsule under alcohol, and tapping on the solid end with a pair of forceps. At first the fuel would come out very fast, then taper off to a thin stream, and finally stop. The procedure would then be repeated. When experience and measurements indicated that all of the fuel had been removed, the inside of the capsule was flushed with alcohol using a 5-cc hypodermic syringe with a 20-gage needle. A clean wooden end of a Q-tip was then worked along the sides and bottom of the fuel cavity. Visual examination of the Q-tip showed no evidence of any fuel remaining.

The fuel was now in two beakers of alcohol. While this procedure was being carried out the screens to be used were washed with alcohol and air dried. They were then weighed on an analytical balance that had been calibrated by the standards and calibration laboratory just a few hours previously in the room it was being used in. The capsule was placed in a 100-ml graduate and bagged out for analysis by gamma scanning. The beakers containing the alcohol and fuel were then bagged out of the enclosure. The plastic bag used to transfer the capsule, forceps, Q-tips, and other tools used in opening and emptying the capsule were then bagged out of the enclosure and canned for analysis by gamma scanning. The plastic enclosure was then taken down, collapsed, and canned for analysis by gamma scanning. The air samples from the plastic enclosure were removed for counting.

The entire inside of the fumehood was lined with plastic and an air sampler head was placed near the exhaust. A 44 μ m screen (44A) was fastened into a holder and placed on top of a clean 1000-ml Pyrex beaker. The alcohol containing the fuel was transferred to this screen using a 25-ml Saebolt pipet. The pipet was rinsed periodically with clean alcohol to prevent any buildup of material. When one of the original beakers was empty a small amount of alcohol was added and a rubber policeman used to scrub the sides and bottom of the beaker. This was transferred to the 44 μ m screen by pouring and simultaneously washing with alcohol from a plastic squeeze bottle. This same procedure was used for the second beaker containing material. The screen, which now contained material <44 μ m plus some material <44 μ m, was placed in a 1000-ml beaker with sufficient alcohol to cover the material. This was placed, by hand, in an ultrasonic tank and agitated for 5 min. The screen was then removed from the beaker and washed with alcohol. Visible fines were evident. The screen was then placed in a crystallizing dish and again the material was just covered with alcohol. This was placed, by hand, in the ultrasonic tank and agitated for 6.5 min. Again fines were visible. These were transferred to a beaker by pouring and flushing with alcohol from a plastic squeeze bottle. This process was repeated five more times. At this point Mr. Hudson, Sandia, was afraid we might be breaking up large particles and suggested stopping the ultrasonic cleaning. The

material was then washed with 100 ml of alcohol poured through the material and screen. This wash was sampled by placing one drop on a piece of filter paper, counting it on a PAC-1SA and calculating the counts per sphere. The equation used is

$$\text{counts per sphere} = \frac{20 \times \text{volume of alcohol (ml)} \times \text{counts}}{\text{number of spheres}}$$

(assume number of spheres to be 80,000/g). We assumed that there were 3 g of spheres. This was done approximately six times with the counts per sphere varying between 400 and 1000.

The screen containing particles $>44 \mu\text{m}$ was placed in a pan and covered with a lid and set aside. The three beakers containing alcohol and fines were placed on a hot plate and the alcohol evaporated to a manageable level. The alcohol and fines were screened through a $20\text{-}\mu\text{m}$ screen and the beakers cleaned in the same manner as before. The screen was placed in a crystallizing dish and ultrasonically agitated. This was repeated several times. The screen was washed once more by pouring some alcohol over the material. Nothing visible came through the screen. This material, $20\text{-}44 \mu\text{m}$ range, was washed out of the screen, into a small plastic bottle, by using alcohol in a plastic squeeze bottle. The screen was set aside to air dry for later weighing. The plastic bottle was placed in a metal can with a friction lid and transferred to analytical. The fines were in the plastic bottle for no more than 15 min. More alcohol was evaporated and then put through the $10\text{-}\mu\text{m}$ screen and washed in exactly the same way as the $20\text{-}\mu\text{m}$ screen. The beakers were cleaned in the same manner as before. The alcohol containing the $<10\text{-}\mu\text{m}$ material was evaporated to approximately 900 ml and the sample taken for the Tracerlab analysis. This sample consisted of one drop of the well-stirred alcohol placed on a microscope slide that had been supplied by Tracerlab. This was repeated for 10 slides. The procedure has been used for all previous capsules and is the same as Tracerlab uses for mounting all Sandia samples they do. The alcohol was then evaporated to approximately 50 ml and transferred to analytical personnel in the same manner as the other samples.

After the beakers and other equipment used for one size range were empty or finished with they were placed in plastic bags, marked with the size range and set aside for analysis by gamma scanning. After the $<10\text{-}\mu\text{m}$ material and equipment were removed from the fumehood, the ultrasonic tank was bagged and removed to make room for the balance.

The weighed screens were stacked and the $>44\text{-}\mu\text{m}$ material transferred from screen 44A to the $177\text{-}\mu\text{m}$ screen. To remove as much material as possible an artists brush was used to clean the screen. The stack with the pan on the bottom and the lid on top was shaken by hand to separate the material into the various size ranges. The 44A screen and pan were weighed together prior to placing the material in the stack. After the screening was complete, each screen, with material, was weighed. The material was then transferred to preweighed primary containers. The

screens were weighed to determine the amount of material held up in the openings. The primary containers and material were also weighed. After all screens were emptied, the pan was weighed to determine the amount of fines that had come off the $>44\text{-}\mu\text{m}$ particles during screening.

After the last step was completed the plastic lining in the fumehood was removed, canned, and gamma scanned.

The material sent to analytical was transferred to 250-ml Teflon beakers and boiled to near dryness. About 25 ml of concentrated HNO_3 was added to the sample and the sample boiled for a few minutes to decompose any remaining organic material which might have been present. A few drops of HF were then added and the sample boiled until dissolution was completed. The sample was then diluted 50 to 1 and counted by liquid scintillation counting. This method is accurate to $\pm 1\%$.

Gamma scan results have an accuracy of $\pm 20\%$. Balance weights were accurate to $\pm 0.5\%$. The gamma scan results were prorated across the range they covered based on the weight of material in the respective size range. For example, the material in the $0\text{-}>177\text{ }\mu\text{m}$ range was prorated for all size ranges whereas material in the $0\text{-}20\text{ }\mu\text{m}$ range was prorated for the two smallest ranges only.

Table I gives the weight distribution for all size ranges and source of weights. Based on the initial sample weight, there was a total of 6.088 g of oxide to be accounted for. All weights were converted to oxide weight before determining totals and calculating percentages. The final calorimeter value (2.4375 W) and the initial weight of fuel (6.088 g) were used to calculate the conversion factor for calculating the oxide weight for the samples assayed by scintillation counting. As Table I shows a total of 6.1312 g were accounted for.

Table I

DISTRIBUTION OF FUEL FROM CAPSULE #15 BY PARTICLE SIZE

	<u>Size Range (μm)</u>	<u>Source</u>	<u>Amount (g)</u>	<u>Total (g)</u>	<u>% of Total</u>
>44 μm	>177	retained on 177 μm screen	0.3824 ¹		
	149-177	retained on 149 μm screen	0.3998 ¹		
	125-149	retained on 125 μm screen	0.5438 ¹		
	105-125	retained on 105 μm screen	0.3486 ¹		
	74-105	retained on 74 μm screen	0.5994 ¹		
	44-74	retained on 44B μm screen	0.7013 ¹		
	44-74	retained on 44A μm screen	<u>0.0532¹</u>		
		(see Table II)	0.0538	3.0823	50.27
20-44 μm		removed from 20- μm screen	1.2681 ²		
		retained on 20- μm screen	<u>0.0153¹</u>		
		(see Table II)	0.0294	1.3128	21.41
10-20 μm		removed from 10- μm screen	0.9022 ²		
		retained on 10- μm screen	<u>0.0203¹</u>		
		(see Table II)	0.0338	0.9563	15.60
<10 μm		passed thru 10- μm screen	0.7268 ²		
		γ scan	0.0253		
		air samples	<u>0.0003</u>		
		(see Table II)	0.0277	<u>0.7798</u>	<u>12.72</u>
				6.1312	100.00
Summary of Table I					
	Total accounted for by discrete size ranges			5.9865g	97.64
	Total prorated (see Table II)			<u>0.1447g</u>	<u>2.36</u>
				<u>6.1312</u>	<u>100.00</u>

¹ by weight² by scintillation counting

Table II gives the amount of material prorated to each size range from samples containing material from mixed ranges. The vertical columns give the total added to each size range.

Table II

FUEL FROM CAPSULE #15 IN MIXED RANGES

Size Range of Fuel (μm)	Proration by Size Range				Total
	<u>>44 μm</u>	<u>20-44 μm</u>	<u>10-20 μm</u>	<u><10 μm</u>	
0->177	0.0538g	0.0228g	0.0164g	0.0134g	0.1064g ^a
0-44	-	0.0066g	0.0048g	0.0039g	0.0153g ^b
0-20	-	-	0.0126g	0.0104g	0.0230g ^c
	0.0538g	0.0294g	0.0338g	0.0277g	0.1447g

^a 0.0044 of this total was estimated from
wipes, remainder by γ scan

^b by weight in pan after screening >44 μm
sizes

^c by γ scan