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SUMMARY OF INVESTIGATIONS UPON THE UO_2 -NaK SLURRY
AS A POTENTIAL REACTOR FUEL

by

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SUMMARY OF INVESTIGATIONS UPON THE UO_2 -NaK SLURRY AS A POTENTIAL REACTOR FUEL

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ABSTRACT

This report summarizes studies carried out with the UO_2 -NaK slurry system to ascertain its potential properties as a fuel for advanced reactor systems. Included are the studies of the physical chemical properties of the slurry, the early loop investigations, in-pile studies, and the exploration of the engineering system behavior of the material. The work was carried out in three different institutions. The Argonne National Laboratory was responsible for the early loop studies and the in-pile work, as well as some phases of the physicochemical behavior. The engineering studies were carried out for ANL by the Mine Safety Appliances Corporation, Callery, Pennsylvania, under Contracts 31-109-38-481 and 31-109-eng-38. The physicochemical studies were performed by the Armour Research Foundation, Chicago, Illinois, sponsored by ANL under Contract 31-109-eng-38-897.

The studies under the program were relatively modest, but they did serve to demonstrate some of the attractive properties of the material as well as its limitations. Specifically, the dropout behavior was demonstrated in piping and tank systems in both conventional and specially designed systems. Hydraulic characteristics were demonstrated, and it was observed that a 10 v/o slurry was the maximum concentration that could conveniently be handled in conventional engineering systems. In-pile performance of the slurry was demonstrated and it was concluded that slurries do have potential for application in specialized, advanced systems.

I. INTRODUCTION

The desirable characteristics associated with a system in which finely divided fissionable material suspended in a liquid metal coolant prompted the initiation of a study program to see if such a system would be applicable for use in a reactor. In particular, if the concentration of the suspended material could be made high enough, such a system

could be used profitably in fast reactors. As a consequence of this reasoning, a program was initiated at the Argonne National Laboratory in the study of the UO_2 -NaK slurry.⁽¹⁾ Following the investigation of the rheological and chemical behavior of the combination, further inquiry into the engineering behavior was performed. These studies were conducted at ANL⁽²⁻⁴⁾ and at the facilities of two laboratories contracted to ANL: the Mine Safety Appliances Corporation⁽⁵⁾ and the Armour Research Foundation.⁽⁶⁾ It is the purpose of this report to describe the studies conducted and the results of these studies.

A. Requirements of Slurries

In order to be considered as a potential reactor fuel, a slurry must fulfill the following basic requirements:

1. The components of the slurry must be chemically stable with respect to one another and the container material. The dispersed phase must be insoluble in the vehicle, for even slight solubility may cause particle deformation or growth.

2. The particles of fissionable material should be of a size such that: a large enough volume fraction can be obtained to make a critical reactor core feasible; that the slurry can be circulated and made homogeneous with a modest expense of power and can be suspended easily after settling; and that comminution of the particles due to thermal stresses, fission recoil, and erosion will be a minimum.

In addition to the above properties, the slurry must maintain a stable concentration when circulating in the reactor system, and the hydraulic characteristics must be such that difficulty is not experienced in pumping and handling.

A slurry of UO_2 in NaK or sodium fulfills some of these basic requirements and, as such, presented the first logical choice to be used in the program of study.

II. INITIAL INVESTIGATIONS

The first investigations of UO_2 -NaK slurries at this Laboratory were made by Abraham and Flotow.⁽¹⁾ A study of slurries of uranium compounds in NaK was initiated with a goal of developing suitable slurry fuels for a fast power breeder and/or thermal reactor. The first compound chosen to study was UO_2 because it was readily available and thermodynamically the most promising.

Table I shows the free energy changes for the reactions of UO_2 and PuO_2 with sodium at three temperatures.⁽⁷⁾ It can be seen that even at the highest temperature neither oxide will be reduced to the metal. The data for silicides, carbides, and nitrides were not complete enough at that time to make worthwhile estimates.

Table I

FREE ENERGY CHANGES (kcal) FOR THE REACTION
OF UO_2 AND PuO_2 WITH SODIUM

	25°C	725°C	1225°C
$\text{UO}_2 + 4\text{Na} = \text{U} + 2\text{Na}_2\text{O}$	67	87	122
$\text{PuO}_2 + 4\text{Na} = \text{Pu} + 2\text{Na}_2\text{O}$	58	78	114

Abraham and Flotow showed that properly prepared UO_2 can be readily suspended in NaK; that low concentrations could be easily moved; that the settled density is low; and that the solids can be re-suspended after prolonged settling.

Preliminary experiments to determine the wetting characteristics were made by measuring the settling rates of UO_2 particles in NaK. Since UO_2 is wetted by liquid sodium at 500°C,⁽⁸⁾ a dilute suspension of UO_2 in NaK would be expected to obey Stokes' law at this and lower temperatures when the UO_2 remains wetted. The technique used was to irradiate a sample of UO_2 and then determine the activity distribution of the UO_2 suspended by stirring in a vertical NaK filled tube. This apparatus is shown in Fig. 1. When a small sample of UO_2 was placed in the stirring apparatus with NaK, it could not be suspended at room temperature by stirring. However, when the NaK was heated to 500°C, it was found that the UO_2 was dispersed throughout the NaK. The UO_2 particles used had a particle size less than 5 μ and at least half should have remained suspended for 2.5 min. It was found, however, that the particles settled out before a count could be taken, and it is clear that the particles agglomerated. The UO_2 could be suspended immediately after cooling and after prolonged settling.

UO_2 may take up oxygen to the composition $\text{UO}_{2.08}$ at room temperature and up to $\text{UO}_{2.17}$ at 600°C without a change in crystal structure.⁽⁹⁾ Since this oxygen would react with NaK to form oxides which could agglomerate the UO_2 particles, the UO_2 was examined for oxygen content, and it was found that the formula corresponded to $\text{UO}_{2.03}$ to $\text{UO}_{2.04}$. A hydrogen reduction treatment at 500°C reduced the composition to $\text{UO}_{2.00}$. This reduced material was used to make a 16.2% by volume slurry, 71.3% by weight, which was suspended in the vertical tube apparatus. The particle size of the UO_2 was 43.7% less than 5 μ , 16.6% between 5 and 10 μ , and 39.7% greater than 10 μ . A typical result is given in Fig. 2. Curve I

Fig. 1. Apparatus for Determination of Settling Rate of UO_2 Particles in NaK

Fig. 2
Activity Distribution in Mixing Tube for a 16.2 v/o Slurry

illustrates the activity profile before stirring, Curve II was obtained while stirring, and Curve III after 16 hr of settling. The settled density of the slurry was 2.9 gm/cc, which corresponds to 20% UO_2 by volume. The shape of the curve at the bottom is due to the fact that the collimating slit has a finite width.

A circulating loop was constructed in order to measure the flow required to maintain a uniform suspension. In addition, the power required

Fig. 3. Schematic of First Slurry Loop

to pump NaK and 5 volume percent (v/o) UO_2 slurry and 10 v/o UO_2 were compared, and it was found that a 10 v/o slurry can be maintained uniform at conventional pumping velocities with modest power dissipation. The loop was constructed from one-inch stainless steel pipe, and a canned rotor centrifugal force pump was used to circulate the slurry. A schematic drawing of the loop is shown in Fig. 3. The combined sump and surge tank contained 75% of the loop volume which had a total operating volume of 5 liters.

The UO_2 used in the loop was obtained from Mallinckrodt Chemical Co. The particles consisted of polyhedra, roughly cubic in shape, about $2\ \mu$ in size, which were cemented together into large aggregates. This material was ball-milled for 48 hr with toluene as a lubricant. The particle size of the material as received and after grinding is given in Table II.

Table II

PARTICLE SIZE OF UO_2

Particle Size, μ	<3	3-5	5-7	7-10	>10
UO_2 as received, w/o	7.6	7.4	10.8	19.8	54.8
UO_2 ground, w/o	88.4	9.4	1.8	0.4	0.0

This material, together with 10 gm of irradiated tracer UO_2 , was degassed and reduced with hydrogen before being placed into the loop. The extent of mixing as a function of flowrate was determined by scanning the sump vertically with a radiation counter. The results are shown in Fig. 4.

Studies were continued, with emphasis being placed on the wetting of UO_2 by NaK and the circulation of slurries in small loops at temperatures up to 650°C .

Fig. 4. Mixing in Sump as a Function of Flow Rate

liquid, can cause flocculation if the contact angle between the vehicle and the suspendoid is greater than zero. In an aqueous system, flocculation may be eliminated by adding a wetting agent or suitable ionic compound that establishes an electric charge on the particles which tends to separate them.

The contact angle between liquid metals and ceramics is seldom zero, and it is clearly not possible to put a charge on the surface of the particles. Therefore, the condition of the surface becomes very important, and very slight changes in the composition can make the difference between wetting and non-wetting. Measurements of the contact angle of sessile drops of NaK on UO_{2+x} plaques were made by Abraham, Flotow, and Carlson,⁽²⁾ who found that at 25°C the contact angle was approximately 110° but dropped to 20 – 22° at 500°C . When the plaques were preheated with hydrogen (surface $\text{UO}_{2.01}$) at 500°C , the contact angle at 25°C dropped to 55° , and at 500°C it was again 20 – 22° . Photos of these sessile drops are shown in Figs. 5 and 6.

A similar situation was found to be true with UO_2 powders. If the UO_2 was outgassed in vacuo at 500°C , then covered with NaK at room temperature, one could centrifuge the NaK cleanly from the solid so that only the dry powder remained. If, on the other hand, the UO_2 was reduced with

A. Wetting of UO_2 by NaK and Na

To suspend a powder in a fluid it is absolutely necessary for the solid to be wetted by the fluid, i.e., the contact angle must be less than 90° . The most desirable situation is to have the contact angle equal to zero, for then the work of adhesion (the energy required to separate the liquid from the solid) is a maximum. Even though this last condition is fulfilled, the powder may still be flocculated⁽¹⁰⁾ and is invariably flocculated if the angle is greater than zero.⁽¹¹⁾ Also, Woodrow, Chilton, and Hawes⁽¹²⁾ have shown that the presence of a third phase, such as a gas or an immiscible

hydrogen at 500°C, the NaK could not be centrifuged away from the powder.⁽¹⁾ Chemical analyses of the UO_2 shows that in the first instance the composition was $\text{UO}_{2.04}$ and in the other, in which wetting took place, the composition was $\text{UO}_{2.01}$. Just by removing a slight amount of oxygen it was possible to achieve wetting.

Fig. 5. Sessile Drop of NaK on UO_2 Plaque at 25°C

Fig. 6. Sessile Drop of NaK on Reduced UO_2 Plaque at 25°C

In the preceding discussion and in the experiments to be described, the effects of Na_2O in a slurry system are shown to be detrimental to the suspendability of the slurry.

Abraham, Flotow, and Carlson⁽²⁾ and Huebotter and Sietz⁽¹³⁾ have shown that Na_2O causes an increased flocculation and "drop out" of suspension at temperatures above 450°C, and that the removal of this oxide by gettering (with uranium metal powder) or cold trapping allows the slurry to be suspended above this temperature.

Abraham, Flotow, and Carlson⁽²⁾ have also offered the following explanation for this effect: the increased flocculation at a high temperature is caused by an increase in the interfacial energy between $\text{UO}_{2.00}$

and NaK. Since the slurry can be suspended at a lower temperature, it is indicated that a soluble impurity or an absorbable one that can be accommodated more easily on the $\text{UO}_{2.00}$ surface at high temperature increases the interfacial tension. The antifluorite structure⁽¹⁴⁾ of Na_2O has a lattice constant of 5.55 Å, and the fluorite structure of $\text{UO}_{2.00}$ has a lattice constant of 5.4704 Å.⁽¹⁵⁾ The UO_2 lattice will accommodate increasing amounts of oxygen as the temperature is raised: at room temperature one can have $\text{UO}_{2.08}$, whereas at 600°C the composition may be $\text{UO}_{2.17}$ without changing the crystal structure.⁽⁹⁾ If it is assumed that the Na_2O is absorbed in the $\text{UO}_{2.00}$ surface with the sodium occupying the oxygen positions, the oxygens will be oriented toward the NaK. This will increase the interfacial energy and cause an increase in flocculation; as the temperature is lowered, less Na_2O can be accommodated and will migrate to the point of contact between UO_2 particles. This will allow the flocs to be broken down into smaller units.

To test this explanation, tellurium metal was added to a small loop. Tellurium is miscible in all proportions with sodium⁽¹⁶⁾ and forms a compound, Na_2Te , which also has an antifluorite structure.⁽¹⁴⁾ The effect was an improvement in the suspendability of the slurry at temperature of 650°C. This behavior is consistent with the idea that the tellurium oriented toward the NaK and kept the interfacial energy at low value.

The toluene-ground UO_2 , which was the same type of material used in the initial experiment conducted by ANL,⁽¹⁾ was analyzed by Armour for impurities and the results are shown in Table III. The interesting data here showed that a large amount of gas and water evolved above 500°C. This accounts for the fact that UO_2 used in the early loop experiment was not free of excess oxygen because the NaK would react with the water to form Na_2O and H_2 . The NaK would also react with CO_2 to form Na_2CO_3 .

Table III

IMPURITIES PRESENT IN TOLUENE-GROUND URANIUM DIOXIDE

(By Mass Spectrometry)

Temperature Range	20-110°C	110-300°C	300-500°C	500-800°C
N_2	-	0.0%	1.0%	0.8%
CO	-	3.8	6.7	0.5
CO_2	-	93.1	89.3	97.7
H_2	-	0.5	2.7	0.0
CH_4	-	0.2	0.0	0.0
Toluene	-	0.7	0.3	0.1
Nitrogen Oxide	-	1.7	0.0	0.9
		100.0%	100.0%	100.0%
Total Evolved gas, cc/g, dry		0.32	0.22	0.50
Free water, %		20.1	0.6	16.6
Total gas cc/g		0.40	0.234	0.60

III. SMALL HIGH-TEMPERATURE LOOPS

A slurry of UO_2 -NaK has been circulated in small loops at temperatures up to 650°C to obtain information on suspendability, erosion, corrosion, and flow characteristics. These loops had a test volume of 133-350 cc and were $\sim\frac{1}{2}$ -in.-ID tubing of Type 304 or 347 stainless steel. The slurries were circulated by electromagnetic pumps. Some of these loops have been described elsewhere, so only brief descriptions and summaries will be given.

A. MARK I

The MARK I loop⁽³⁾ shown in Fig. 7 was fabricated in the shape of a parallelogram 10 in. x 18 in. The UO_2 used in this loop was the ground Mallinckrodt UO_2 , and the particle size is given in Table II. This UO_2 was treated with hydrogen at 500°C so that the O/U ratio was 2.01. Enough UO_2 was added to the loop to make a 4.3 v/o slurry. The only measurements made with the slurry were composition changes as a function of flowrate and temperature. Composition variations were monitored by observing the changes in counting rate from a counter, shown at right in Fig. 7.

Fig. 7. MARK I Slurry Loop

The measured gamma activity was obtained from UO_2 in the loop, part of which was irradiated with neutrons prior to insertion into the loop. Figure 8 shows a plot of counting rate versus flowrate. The plateau region on the curve was taken as an indication that all the UO_2 was in suspension when the flowrate was above $2\frac{1}{2}$ ft/sec. During the course of

Fig. 8. Counting Rate as a Function of Flow Rate at 25°C in MARK I Slurry Loop

the experiment, it was observed that, as the temperature of the slurry exceeded 500°C, the flowrate dropped from any fixed value in the range of 3-8 ft/sec to as low as 1.3 ft/sec in a matter of minutes. At the same time, the counter indicated no UO_2 in suspension. As soon as the temperature was lowered to 480°C, the flowrate returned to the higher figure and the UO_2 was completely suspended in a matter of seconds. This drop in flowrate was believed to be due to increased flocculation at the higher temperature, which caused the UO_2 to drop out of suspension.

B. MARK II

The MARK II loop⁽³⁾ was constructed to clarify the question of composition uniformity and to try to eliminate the flocculation at high temperatures. A schematic diagram of MARK II is shown in Fig. 9. The pressure taps located on the right vertical section were incorporated in the loop to measure slurry composition. The pressures were measured by forcing the fluid in the pressure tubes down to a reference mark with purified helium gas. The location of the liquid level was detected by an induction method similar to that described by Pulsford.⁽¹⁷⁾ The radiation monitor described in the MARK I loop was also used.

In an effort to remove any possibility of oxygen contamination being introduced with the UO_2 and to reduce the impurity level, the UO_2 for this loop was heated in NaK at 800°C for 7 hr, the NaK destroyed with N-butyl alcohol, and the UO_2 washed free of alkali and dried. The material was given a final treatment with hydrogen at 600°C before introduction into the loop. The O/U ratio of this material was found to be 2.003 ± 0.003 .

Densities were measured at room temperature for NaK Slurry I (1.6% UO_2) and Slurry II (4.0% UO_2). The density measurements made with these slurries showed that the assumption made for the plateau region in the

Fig. 9. Schematic of MARK II Slurry Loop

counting rate versus flowrate plot (see Fig. 8) in the MARK I experiment was correct; that is, all the UO_2 was in suspension. Despite the care taken to eliminate oxygen contamination, this loop showed the same temperature effect as MARK I. However, a small addition of powdered uranium metal stabilized the slurry at temperatures up to 600°C .

C. MARK III

A schematic diagram of the MARK III loop is shown in Fig. 10. The essential feature of this loop was provision for making pressure measurements so that viscous losses and density of the circulating fluid could be determined.

Fig. 10. Schematic of MARK III Slurry Loop

The loop measured approximately 21 in. square with a circulating volume of 222 cc. The pressure taps in the horizontal and vertical legs were spaced 12 in. apart and were determined to be hydrodynamically identical up to flowrates of 7 ft/sec. Measurements were made with differential pressure transmitters.⁽¹⁸⁾ The particle-size distribution, as determined in water, showed that 90% were less than $10\ \mu$ and 25% less than $2\ \mu$. The UO_2 was treated with hydrogen, as in previous experiments, prior to adding it to the loop. The Pseudo-Shear diagrams plotted from the data taken on this material are shown in Fig. 11.

It should be noted here that the consistency of 5.09 v/o slurry changed after only 5 hr of heating. The curve indicates that the slurry became more flocculated so that the fluidity approached that of NaK, and the flocs did not break down even at the highest rates of shear. The pressure taps on this loop plugged after 50 hr of operation, but gross changes in the slurry were monitored with the flowmeter and radioactivity counter. This loop also exhibited the "drop out" behavior, and suspension was resumed at the high temperatures by the addition of uranium metal powder.

Fig. 11. Pseudo-Shear Diagrams of UO_2 in NaK: (A) at 50°C and (B) at 550°C

To test the hypothesis that Na_2O was responsible for flocculation, oxygen was added to the loop in the form of Na_2O_2 . The flocculation increased to the point where flow almost stopped. When the temperature was lowered to 500°C , the UO_2 completely resuspended. Uranium metal powder was again added, and the condition of the loop returned to the same as before the Na_2O_2 addition. This loop was also used for the tellurium experiment referred to previously.

D. MARK IV

A small MARK IV loop which measured 18 by 6 in. was fabricated to circulate a 15 v/o slurry. The slurry was sufficiently fluid to be circulated in the apparatus and appeared to be suspended uniformly.

E. MARK V

A MARK V loop which measured 24 by 48 in. was fabricated from $\frac{1}{2}$ -in.-ID by $\frac{5}{8}$ -in.-OD tubing. This loop incorporated a density-measuring section, a reversible pump, a hydrocyclone, and a valve arrangement with movable check balls. A schematic diagram is shown in Fig. 12 (taken from unpublished data of Flotow).

F. Erosion and Corrosion Test Loop

Previous studies of the circulation of UO_2 -NaK slurries at high temperatures had all been of short duration. To determine the extent of erosion of the conduit and comminution of slurry particles at a temperature of 600°C and a flowrate of 6 ft/sec, a 5 v/o UO_2 slurry was circulated for 13,000 hr in a prototype in-pile loop. During the course of the experiment, X-ray photos were taken of the loop to verify that the slurry was

circulating. These photos show the flocculated nature of the stagnant slurry and the effect of centrifugal forces on the slurry particles at the bends in the loop. As previous experiments have shown, the slurry could not be circulated above 500°C without the addition of an oxygen getter, such as uranium metal. Metallographic examination at the termination of the experiment showed little evidence of erosion or corrosion of the stainless steel loop, nor was there appreciable comminution of the UO_2 particles.

Fig. 12. Schematic of MARK V Slurry Loop

The loop was constructed from $\frac{1}{2}$ -in.-OD, Type 304 stainless steel tubing with a 0.020-in. wall. The tubing was bent to form a close 180° bend at the top and bottom of the loop. A dc permanent magnet pump was used to circulate the slurry in the loop at flow velocities up to 10 ft/sec. The expansion tank was located on the top bend of the loop and contained a gas cover of helium. The flow velocity was measured with a magnetic flowmeter. Temperatures were recorded by chromel-alumel thermocouples welded to the loop.

After welding, the loop was thoroughly cleansed and loaded with 350 cc of NaK (78 w/o K), which was then circulated for several hours at 450°C to insure wetting of the loop, pump, and flowmeter.

The particulate UO_2 for the slurry was the same material used in the MARK I loop and was treated in the same way prior to insertion. The particle-size distribution of this material is given in Fig. 13. One gram of UO_2 was irradiated in the CP-5 reactor and added to the bulk of the UO_2 to act as a radioactive tracer. One hundred and sixty-one grams of UO_2 , enough to make a 5 v/o slurry, were added slowly to the loop while the NaK was flowing, and the occurrence of suspension of UO_2 was determined by scanning the loop with a scintillation counter. In case of a rupture, the loop was sealed inside a containment vessel to prevent the spread of radioactivity.

Fig. 13. Particle Size Distribution of UO_2 Used in Erosion-Corrosion Test Loop

For the first 9000 hr of operation, the loop was operated below 400°C . When the temperature was raised above 400°C , the flow-rate fell rapidly due to the dropout effect from 6 ft/sec to $\frac{1}{4}$ ft/sec. After 9000 hr of operation, 10 gm of powdered uranium metal were added to the slurry and the loop heated to 600°C . The slurry continued to flow without the drop in flowrate previously experienced.

During the course of the experiment, X-ray photos were taken of the loop. Figure 14 shows the flowing slurry in the loop at 600°C . The photo was taken over an exposure time of 3 min at a flowrate of 6 ft/sec. This photo shows that the slurry was circulating as expected and was not stuck to the walls of the tubing.

Fig. 14. X-rays of Dynamic and of Static Slurry in Straight Sections of Erosion-Corrosion Test Loop

Figure 14 also includes a view of the same section of the loop with the electromagnetic pump turned off and the slurry settled out. This photo shows that the slurry was flocculated when allowed to settle. The flocculations were so large that the tubing was actually bridged to support the slurry, and a settled level could not be determined. This would probably not be the case if the tubing were larger in diameter.

In Fig. 15, the slurry depicted was flowing through a bend in the loop and upward in the left-hand tube. The centrifugal force on the dense UO_2 particles had forced the majority to flow along the upper wall of the bend. This photo was taken when the conditions in the loop corresponded to 600°C and a flowrate of 6 ft/sec. Figure 15 also shows the same bend with the pump off and after slurry has settled into the lower part of the bend. One can see the flowing slurry in the lower bend of the loop; note that the UO_2 was flowing along the outer wall of the bend.

After 13,000 hr of operation, the loop failed because of formation of a $\frac{1}{2}$ -in.-diameter hole in the tubing at the welded joint in the lower bend. Metallographic examination of the weld area showed that the region appeared sound. The structure of the tubing and the coupling, as shown in Fig. 16, was that of sensitized Type 304 stainless steel. After dissection of the loop, it was noticed that there were protrusions on the inner edges of the pump tube section. These protrusions appeared to be flared metal resulting from the tube-cutting process before the loop was assembled. Figure 16 shows the flares with stressed metal and carbide precipitation occurring in the tail-like portion of the flare. This was also the structure of sensitized steel. Figure 16 shows an area along the inner wall and shows no evidence of decarburization.

Comparison of measurements of the wall thickness with that of a section of unused tubing revealed no discernible erosion effects. The average of four readings on the used tubing was 0.0193 in. and on the new was 0.0192 in. Spectrographic analysis of the UO_2 at the termination of the experiment showed the presence of 0.6% stainless steel; therefore, the tubing failure was not a result of erosion. In all probability, a small fracture occurred in the weld, allowing NaK to leak out, which corroded the stainless steel when the oxides formed at high temperature on contact with air.

Particle-size analysis of the UO_2 , shown in Fig. 13 after 13,000 hr of circulation, revealed little change in the size distribution.

(A) SLURRY FLOWING THROUGH A BEND

(B) SLURRY SETTLED IN SAME BEND

(C) SLURRY FLOWING THROUGH LOWER BEND

Fig. 15. X-rays of Dynamic and of Static Slurry in Curved Sections of Erosion-Corrosion Test Loop

(A)

TUBE AND COUPLING-LOWER BEND

(B)

INNER TUBE WALL

(C)

FLARED METAL IN PUMP TUBE

Fig. 16. Photomicrographs of Loop Tubing

IV. IN-PILE EXPERIMENTS

A. In-pile Prototype

Prior to the fabrication of an in-pile loop, an in-pile prototype was built which was identical with the MARK I-P except that the UO_2 used to make the slurry was the unground Mallinckrodt material with a particle-size distribution shown in Table II. This material of larger particle size was added to the loop to make a 5 v/o slurry. After prolonged settling the material was difficult to re-suspend in the hairpin-shaped loop. The loop was operated for several hundred hours at temperatures up to 450°C . The main purpose of construction and operation of this loop was to gain experience in handling the in-pile experiment. The loop after use was therefore dismantled in a hot cell in order to develop a technique for handling the MARK I-P experiment.

B. MARK I-P

Since it had been established that a UO_2 -NaK slurry can be circulated for an extended period of time at 650°C , that the concentration was uniform throughout the circulating volume, and that the system was not subject to erosion nor corrosion, it was important to the continued development of this program to examine the slurry behavior as fission products were built up in the system. It was thought that comminution of the particles as a consequence of the fission process and/or fission product contaminants might adversely affect the rheology. This section of the paper describes an in-pile loop which was inserted in a vertical thimble of the Argonne CP-5 heavy-water thermal reactor and the observations made while circulating a 5 v/o slurry of 93% enriched UO_2 in this loop. The loop was operated for 902 hr at temperatures above 350°C , with the total heat being generated by the fission process. Analytical results concerning the UO_2 , the NaK, and the cover gas after termination of the experiment are also presented.

The loop was constructed of $\frac{1}{2}$ -in.-OD (0.020-in. wall), Type 304 stainless steel tubing; the height was $3\frac{1}{2}$ ft and the perimeter measured $7\frac{1}{2}$ ft. A schematic drawing of the loop in the containment vessel is given in Fig. 17 and shows the location of the Faraday-type electromagnetic pump, the flowmeter, expansion tank, and boral neutron shield. The dotted lines in the figure represent tubes that were used for circulating air in order to remove the heat. These tubes were in thermal contact with the loop at the location shown by spring-loaded aluminum clamps, which also restricted the motion of the loop in the horizontal plane. The containment vessel was constructed of stainless steel tubing, the upper 2 ft being 5 in. in OD and the lower 3 ft being 3 in. in OD. The upper portion was enlarged to accommodate the pump and flowmeter. Prior to inserting the loop into the reactor, the containment vessel was evacuated to a pressure of 10^{-2} mm Hg in order to reduce the heat transfer to the reactor.

Fig. 17. Schematic of Mark I-P
In-Pile Slurry Loop

The expansion tank located at the top of the loop had a volume of 250 cc, which was equal to the circulating volume of the loop. The valve shown in Fig. 17 was used for adding NaK and helium cover gas to the loop as well as for sampling the gas at the termination of the experiment. A separate port, indicated in the drawing, was used for adding the UO_2 . During the experiment, the loop was charged with 265 cc of NaK and with helium gas at a pressure of one atmosphere at room temperature.

The electrical leads for the flowmeter and thermocouples were brought out of the containment vessel by a hermetically sealed Amphenol connector, not shown in the drawing. The loop was suspended vertically. The pump leads were sealed with stainless steel bellows. These bellows presented a high-resistance path to the current, so that at full voltage

(2.3 v) only 50 w were dissipated in both bellows. This method of suspension had the advantage that thermal expansion could take place without stressing the loop. The pump was designed to fit into the available space, efficiency, in this case, being of little concern. The field coils were wound on a laminated iron core and connected in series with the electrodes, which were silver soldered to the loop tubing. The tubing had been flattened, at that point, to provide a passage of $\frac{1}{8} \times \frac{1}{2}$ -in. cross section. The pump was operated with ac in order to keep the high-current leads as short as possible. Figure 17 shows the transformer cast into the reactor shield plug to eliminate the necessity of running several hundred feet of buss bar to conduct power to the pump. With a power input of 675 w, the slurry could be circulated at a velocity of 9.2 ft/sec.

Figure 18 also shows, schematically, the loop positioned in a vertical thimble in the graphite zone of the CP-5 reactor. Located at the bottom of the containment vessel is a boron, neutron-absorbing shield which could reduce the fission rate by a factor of 100 when the slurry was permitted to settle out of suspension. The incident thermal-neutron flux in the hole had a maximum value of $1.37 \times 10^{12} \text{ n}/(\text{cm}^2)(\text{sec})$, but, after the absorption in the stainless steel and slurry, the actual power from fission as determined from burnup was 1800 w.

Fig. 18. Schematic of Slurry Loop in CP-5 Graphite Zone

The cooling air for the loop was drawn from the reactor room through the tubes indicated as dotted lines in Fig. 17 and 18, and exhausted into a retention room to allow for the decay of A^{41} before passing into the atmosphere. The air flow was adjusted manually, which allowed the loop temperature to be changed within a range of 100°C . An air pressure-actuated switch was interlocked with the reactor scram circuit to shut the reactor down if the cooling air failed.

Thermocouples were located at various positions along the length of the loop. One thermocouple was interlocked with the reactor scram circuit to shut the reactor down if the temperature rose above 500°C . The flowrate of the slurry was measured with a magnetic flowmeter, which was also interlocked with the scram circuit to shut the reactor down if the flowrate dropped below 1 ft/sec.

As was pointed out previously, the presence of Na_2O adversely affects the rheology of the slurry. Care was therefore taken to keep the loop filled with helium gas during the welding operations to reduce oxide formation, since NaK will reduce the oxides formed by stainless steel. After the welding operations were completed, the loop was cleaned with a mixture of nitric and hydrofluoric acids, washed, dried, and then outgassed at 500°C at a pressure of 10^{-5} mm Hg. Finally, 265 cc of NaK (78 w/o K) were filtered into the loop. The NaK was circulated for 16 hr at 400°C to insure wetting of the flowmeter and pump before adding the UO_2 .

The UO_2 (93.14% U^{235}) was obtained from the Union Carbide Nuclear Corporation, Oak Ridge, Tennessee. It was prepared by precipitating ammonium diuranate from a uranyl nitrate solution and then reducing the diuranate in a hydrogen atmosphere at 950°C . After the reduction, the UO_2 was passed through a micro-pulverizer to reduce the particle size. Analysis of the UO_2 showed the principal impurities to be: F 539 ppm; Fe 285; Al 78; Ca 50; Cu 40; Cr 20; and Sn 18. The remaining impurities, consisting of 14 elements, were less than 220 ppm total. The particle-size distribution was determined by a sedimentation technique,⁽¹⁹⁾ and the cumulative distribution is shown as Curve A in Fig. 19. The distribution as shown is not in itself of particular significance, but will be discussed later, as it relates to Curve B in Fig. 19.

Fig. 19. UO_2 Particle Size for MARK I-P In-Pile Slurry Loop

A quantity of UO_2 (131.6 gm), sufficient to form a 5 v/o suspension, was placed in a tube furnace, out-gassed at 600°C , and reduced with hydrogen at 700°C to obtain oxide which approached the stoichiometric composition. The UO_2 was then transferred into the loop through a vacuum lock while the NaK was circulating. When it was established, with a scintillation counter, that all the UO_2 was in the circulating stream, the loop was heated to 450°C for 3 hr to establish the minimum contact angle with the particles, cooled, and inserted into the containment vessel.

Except for short periods of time for testing temperature distribution and rate of temperature rise, the slurry was circulated at an average velocity of 5 ft/sec. When the reactor was at full power (2 Mw) and with 35 cfm of cooling air through the thimble, the loop temperature was 350°C . The loop was operated in the reactor for a total of 960 hr, of which 902 hr were at temperatures above 350°C . It was also operated for short periods as high as 470°C by reducing the air flow. During this period, the loop was thermally cycled 82 times between 350°C and 50°C , the reactor ambient temperature, because of reactor shutdown. From the measurement of the inlet and exhaust air temperatures, the quantity of heat transferred to the air was calculated to be 2300 w, which agrees quite well with the value of 2400 w calculated for the heat generated in the slurry from fission plus the heat generated by the pump.

To establish the fact that the slurry was being circulated around the loop, the pump was turned off during a reactor shutdown and the UO_2 was permitted to settle out of suspension into the neutron-shielded portion of the loop. As was shown in Fig. 17 and 18, the lower quarter of the containment vessel was surrounded by a stationary boral shield which reduced the thermal-neutron flux by a factor of 100. When the reactor was taken up to full power, it was found that the only thermocouple that registered a temperature rise was the one located at the top of the boral shield, which indicated that some neutrons were coming in from the top because of incomplete shielding or that the top of the settled bed was slightly above the shield. When the pump was turned on, oscillation of the flowmeter for the first 30 sec and the rapid temperature rise in the loop indicated that the UO_2 was being suspended.

During the whole period of operation, no complications developed, and within the temperature range tested the slurry presented no mechanical difficulties whatever.

The loop experiment was terminated after 960 hr of operation, when the Argonne CP-5 reactor was shut down for modifications. The loop was transferred to a hot cell in order to analyze the NaK, the UO_2 , the cover gas, and the loop components.

A sample of the cover gas was taken by connecting a 500-cc evacuated bulb to the bellows valve on the expansion tank, shown in Fig. 17. This gas was analyzed by absorbing the krypton and xenon on activated charcoal at liquid nitrogen temperature. The total volume of fission product gases was found to be 0.88 cc (STP), which represented 45.5% of the gas volume expected from the burnup calculated from the cerium fission product analysis. Mass spectrographic analysis of the xenon showed Xe^{131} , Xe^{132} , Xe^{134} , and Xe^{136} to be in the expected ratio.

The loop was vibrated slightly to compact the UO_2 and was then scanned with a radiation counter to locate the level. The loop was then crimped to isolate the UO_2 from the bulk of the NaK. The clear NaK was drained from the loop and reacted with butyl alcohol; the resultant solution will be discussed as Sample 1. The UO_2 was removed from the loop, and the NaK that remained with the solids was also reacted with butyl alcohol. This solution was then combined with the ethyl alcohol and water washings from the UO_2 , and will be discussed as Sample 2. The washed UO_2 was dried, and 329 mg were dissolved in HNO_3 to be analyzed as Sample 3.

The three solutions were analyzed for Cs^{137} , Ce^{141} , and Zr^{95} to arrive at the burnup and location of fission fragments. The results of the analysis are given in Table IV, in which the percentage of each nuclide found in the various fractions is tabulated.

Table IV

RELATIVE PERCENTAGES OF FISSION
PRODUCTS FOUND IN LOOP SAMPLES

Sample	Cesium	Cerium	Zirconium
Solution 1 (NaK fraction)	62.0	0.03	0.12
Solution 2 (NaK wash)	19.8	0.02	0.11
Solution 3 (UO_2)	18.2	99.95	99.77

Part of the UO_2 remaining from the preparation of Solution 3 was leached with concentrated HCl for one hour under a heat lamp in order to determine the quantity of cesium remaining on the surface of the UO_2 . Only an additional 0.65% cesium was removed by this treatment.

One can conclude from the analyses presented in Table IV that at least 82% of the fission fragments were ejected from the UO_2 particles if diffusion of the fission products through the UO_2 is assumed small. Previously reported data⁽³⁾ showed that diffusion of fission products from powdered UO_2 was so slow that it was not detectable even after 300 hr in 800°C NaK. This result is in fair agreement with the calculations of McDole⁽²⁰⁾ and of Gardner,⁽²¹⁾ and with the experimental results of Hermans and Plas⁽²²⁾ on the escape of fission fragments from ThO_2 suspensions. The small particle size of UO_2 ($1.8\ \mu$ average) permits all of the fission fragments to escape from the primary particle, since the range of a fission fragment recoil in UO_2 is approximately $10\ \mu$. The fission fragment may then be stopped in an adjacent particle or in the fluid. The probability of stopping in an adjacent particle is a function of the solids size and concentration. Gardner has shown for slurry particles under $3\ \mu$ and for a concentration of 5 v/o that 10% of the fragments lodge in the particles.

Since it has been demonstrated⁽²⁾ that the minimum contact angle of NaK on UO_2 is approximately 20° , small flocs rather than individual particles were being circulated.⁽¹²⁾ On this basis, it is expected that the percentage of the fission fragments lodged in the UO_2 would be higher than that calculated from single-particle assumptions.

The two atoms of oxygen that are left in the particle after the uranium fission may diffuse out and form Na_2O or a fission product oxide that is stable in sodium. Thermodynamic calculations made by Epstein and Nigriny⁽²³⁾ show that at least 83% of the oxygen, from the fission of U in UO_2 , will form fission product oxides if equilibrium is assumed, and, therefore, there will be a continued buildup of Na_2O in the system. This can be controlled, as previously stated, by adding powdered uranium metal.

In addition, the results in Table II indicate that the majority of the cesium, an example of a fission product soluble in NaK, will be found in solution, whereas cerium and zirconium, examples of two elements and associated oxides insoluble in NaK, will be found on the UO_2 particles. Further information must be obtained, however, before one can predict the phase distribution of all the fission products, because it is not possible to state from a knowledge of chemistry whether the halides and metalloids are to be found in the NaK or on the UO_2 .

Since some losses of solution were incurred during the transfers in the hot cell, only the results from cerium were used to calculate the total burnup. The calculation showed that 0.070% of the uranium had undergone fission. This figure is greater by more than a factor of two than that calculated from the quantity of krypton and xenon collected. With the use of 18% as the value of fission gases that were lodged in the UO_2 particles, it may be calculated that approximately 37% of the fission gases were entrained with the slurry. This result is not at all surprising in view of the contact angle of NaK on UO_2 . The points of contact of the particles will serve as loci for gas entrainment.

Particle-size analysis of the UO_2 after the loop experiment is shown in Fig. 19 on Curve B along with the analysis of the original material. It can be deduced that extensive comminution of the particles occurred. However, the rheology of the suspension was unaffected, probably because of the flocculated nature of the suspension. Measurements of surface area performed at the Armour Research Foundation by the method of Brunauer, Emmett, and Teller,⁽²⁴⁾ showed the original material to have an area of $0.370 \text{ m}^2/\text{gm}$, whereas the post-irradiation sample had a surface area of $0.836 \text{ m}^2/\text{gm}$. The increase in area is easily accounted for by the decreased particle size.

In conclusion, then, it was found that the slurry performed in the reactor very satisfactorily, as it did in previous out-of-pile experiments up to a temperature of 350°C . The fissioning in UO_2 did degrade the particles, but there was no adverse effect on the flow properties of the slurry. Using cesium and cerium as the examples of a soluble and insoluble element, respectively, one concludes that those fission fragments soluble in NaK will be found in the NaK and those insoluble in the NaK will be found on the UO_2 . When the loop was dismantled in the hot cell, no evidence of UO_2 adhering to the walls of the loop was found. After 902 hr at temperatures above 350°C , the presence of fission products on the UO_2 did not have an adverse effect on the circulation of the slurry.

C. MARK II-P Loop

MARK II-P loop was designed to obtain information in the 15 v/o UO_2 range at higher temperatures. The rheology of the slurry under irradiation was to be determined by measuring the pressure drop of the flowing fluid in an upward flowing leg and downward flowing leg. The density was continuously measured by using pressure taps as a density bridge. For the upward flowing stream, $\Delta P_{\text{upward}} = \Delta P_{\text{flow}} + \Delta P_{\text{density}}$, and for the downward flowing stream, $\Delta P_{\text{downward}} = \Delta P_{\text{flow}} - \Delta P_{\text{density}}$. Therefore,

$$\frac{1}{2} (\Delta P_{\text{up}} - \Delta P_{\text{down}}) = \Delta P_{\text{density}}$$

and

$$\frac{1}{2} (\Delta P_{\text{up}} + \Delta P_{\text{down}}) = \Delta P_{\text{flow}} \quad .$$

MARK II-P was constructed from $\frac{5}{8}$ by $\frac{1}{2}$ -in. Type 304 stainless steel tubing formed in a hairpin shape. The loop was 5 ft in height and had a circulating volume of 334 cc. A schematic diagram, Fig. 20, shows the loop in the containment vessel with an associated photo showing the loop without the containment vessel. These figures show the positions of the pump, pressure transducers, expansion tank, heat exchanger, and flowmeter.

Figure 21 shows a schematic diagram of the transducers. The diaphragms, 0.005 in. thick, were purchased from the Foxboro Company. The linear variable differential transformer (LVDT) (Automatic Temperature Control Company, Philadelphia, Pa., Type 6208A) had a linear range of ± 0.15 in. and output of 0.18 mv per exciting volt per 0.001-in. armature motion. The indicator, obtained from the same company (multichannel indicator class 6103A), had a linear scale subdivided into 1000 divisions; each division was equivalent to approximately 0.25 mm Hg pressure. Three-volt, 60-cycle ac was used to excite the LVDT in the pressure gauge and the matching transducer in the indicator. The pressure gauge was not thermally compensated because the spring constant of the diaphragm would change with temperature; therefore, an effort was made to keep the gauge at a temperature below 100°C.

The pressure sensitivity of these units was 0.25 mm Hg, and the accuracy was $\pm 0.5\%$ over the calibrated range from 50 to 250 mm. The transducers were connected to the two pressure taps on each leg: one transducer recorded the ΔP of the upward flowing slurry, and the other ΔP of the downward flowing slurry. Figure 20 shows the taps (in each leg, which were 2 ft apart) connected to the transducers. The lines at lower pressure were in thermal contact with the loop up to the upper taps, so that the NaK temperature in the pressure tube and the slurry were the same. From there the lines were in thermal contact with each other so that the temperature gradient between the loop in the transducer would be the same in each line.

The electromagnetic pump used with this loop was a series-wound Faraday type. Power was supplied to the pump by a transformer located in the shield plug. The pump was capable of circulating NaK at a flowrate up to 20 ft/sec and 15 v/o slurry up to 9 ft/sec.

Fig. 20. MARK II-P In-Pile Slurry Loop

Fig. 21. Schematic Pressure Transducer
Used on MARK II-P In-Pile
Slurry Loop

the experiment, the fluid was monitored and very little activity was found. In the filter, however, the activity was about 10 mr/hr, which showed that small particles, perhaps copper oxide, were being trapped in the filter.

A movable boral shield (see Fig. 20) was placed around the lower section of the loop and used to adjust the heat generation rate in the loop.

The loop was carefully cleaned with a mixture of nitric and hydrofluoric acids, washed, dried, and outgassed at 500°C at a pressure of 10^{-5} mm of Hg. Finally, 350 cc of NaK (78 w/o potassium) were filtered into the loop. The NaK was circulated at temperatures up to 650°C to insure wetting of the loop, pump, and flowmeter. The loop was heated by passing a high current, 700 amp at 2 v, through the loop, which generated heat in the loop and NaK. A check on the calculated thermal resistance of the heat exchanger was made at this time by circulating the cooling fluid (MIPBP). To check out the pressure taps, data were taken on the pressure drop due to flow of NaK at room temperature; these data, shown in Fig. 22 agreed quite well with previous data.⁽²⁾

The UO_2 powder obtained from ANL Metallurgy and prepared by the reduction of U_3O_8 with hydrogen was ground in a ball mill to the proper particle size. Figure 23 shows the cumulative particle-size distribution of the UO_2 before irradiation as determined by a sedimentation technique.⁽¹⁹⁾ Analysis of the UO_2 showed the principal impurities to be: Al 100 ppm; Cr 2 ppm, Cu 8 ppm, Fe 60 ppm, Mg 66 ppm, Mo 30 ppm, Na 10 ppm, and Si 1000 ppm. The isotopic composition was as follows:

$$\text{U}^{234} = 1.03\%; \text{U}^{235} = 93.24\%; \text{U}^{236} = 0.484\%; \text{U}^{238} = 5.25\%.$$

The heat produced by the fission process was removed by a circulating organic fluid, mono-isopropyl biphenyl (MIPBP). The heat was transferred from the slurry to the fluid through a heat exchanger that clamped to the loop. This clamp was so constructed that it allowed the tubing to expand due to the increase in temperature with respect to the cooling tube without undue stress in the loop. The fluid was pumped from the reactor thimble down to the basement, where it passed through a water-cooled heat exchanger, and was recirculated. Copper and stainless steel were used in the cooling system piping, and a filter was placed in the system to remove circulating particles that would become radioactive because of neutron irradiation in the reactor. During

Fig. 22. Pressure Drop in MARK II-P Slurry Loop at 50°C

Fig. 23. UO_2 Particle Size for MARK II-P In-Pile Slurry Loop before Irradiation

Four hundred and thirty-seven gm of UO_2 , a quantity sufficient to make a 11.04 v/o slurry, were placed in a tube furnace and heated to 600°C, outgassed, and reduced with hydrogen at 700°C to form an oxide that approached a stoichiometric composition. The UO_2 was then transferred into the loop through a vacuum lock while the NaK was circulating at a velocity of 4 ft/sec. A scintillator was used to determine that the UO_2 was being suspended.

A second batch of UO_2 , 66 g, was treated in the same manner, and 10 g of uranium metal in the form of $\frac{1}{8}$ -in. rod, $\frac{3}{8}$ in. long were added to the UO_2 . The uranium metal was powdered by preparing the decomposing uranium hydride in the tube furnace. This UO_2 and uranium metal powder mixture was then added to the loop. After this addition, the flowrate fell almost to zero, and it was thought that some of the uranium powder had sintered together and formed small lumps that had lodged in the electromagnetic pump tube. To prove this, a 0.020-in. by $\frac{1}{4}$ -in. piece of spring stock was inserted through the UO_2 -loading port, into the tee for the expansion tank, and the pump flow was reversed. The lumps were stopped in the tee and X-ray pictures (see Fig. 24) show the four, small, sintered uranium lumps. Next, a small screen basket was inserted into the tee and the pump turned on. After a few trials, the lumps were deposited into the basket and removed from the loop. Before the UO_2 -loading port was pinched off and welded, the uranium metal removed from the loop was replaced with powdered uranium metal, and the pressure in the expansion tank was adjusted to one atmosphere. The loop was placed in the containment vessel, which was then welded shut, helium leak tested, evacuated, and sealed off.

The loop was placed into a vertical thimble of the Argonne CP-5 reactor. The reactor was brought up to power, and the coolant flow and

Fig. 24. X-ray of Uranium Lumps Lodged in Tee of Loop

the boron shield were adjusted so that the temperature of the loop was 200°C . It was intended to run at this temperature for a period of 2 weeks, before increasing the temperature and thermal cycling to take data.

From the coolant flowrate and the temperature change ΔT , the amount of power released from fission was calculated to be 2500 w. There were, however, some heat losses to the reactor which were difficult to estimate.

After one week of apparent normal operation, the loop scrambled the reactor because of low flowrate: neither the flowmeter nor the pressure transducer registered flow.

Various checks were made to deter-

mine the cause, and the conclusion was that either a rupture in the tubing or a solid plug had formed. The apparatus was therefore removed from the reactor. The activity at the time of removal was >500 r/hr at 1 ft.

After being transferred to a hot cell, the containment vessel was removed in order to examine the loop. The tubing appeared sound and there was no evidence of leakage. If a plug had formed, the most probable place would be in the pump tube, because the restriction in the pump tube was $\frac{1}{8}$ in. $\times$ $\frac{5}{8}$ in. in cross section. The pump tube was cut out and the UO_2 found in it examined. Some $\frac{1}{16}$ -in.-diameter lumps were found, but they could be broken up with slight pressure. Although the evidence was inconclusive, a group of such lumps could have caused a plug. It is believed that all the uranium metal lumps were not removed from the loop, and that they finally collected in the pump tube causing the plug.

Figure 22 shows the pressure-drop data for an approximately 15 v/o slurry at 50°C and compares it with previous data⁽²⁾ at 5 v/o at 50°C .

The density data for this slurry were not as satisfactory as anticipated. There was difficulty in repeating the density measurements at various flowrates. X-ray pictures of the expansion tank and pressure tap tubes show that all the UO_2 was not in the circulating stream and that some of the slurry was suspended in these noncirculating portions of the loop. Pressure-drop data show that the actual concentration in the circulating volume varied from 13.5 to 11.5 v/o UO_2 . Figure 25A shows the slurry circulating, and Fig. 25B shows the slurry settled.

Fig. 25. X-rays of (A) Dynamic and (B) Settled Slurry in MARK II-P In-Pile Loop

Although the experiment was terminated because of a low-flowrate condition, some information was obtained to help design a future experiment. Although a plug occurred, it was not because of the UO_2 slurry, but because of the sintered uranium metal. It is essential that the uranium metal additions be in very finely divided powder. The experiment showed that a 13.5 v/o slurry could be circulated and that a higher concentration could probably be circulated if the particle size were increased.

D. MARK III-P

A MARK III-P in-pile loop was designed taking advantage of the experience gained from the MARK II-P experiment. A photograph of the loop is shown in Fig. 26.

The loop was constructed from $\frac{5}{8}$ -in.-OD by $\frac{1}{2}$ -in.-ID, Type 304 stainless steel tubing and had a circulating volume of 490 cc. The pressure-tap design was improved so that the lines did not plug with slurry. Figure 27 shows the construction of the pressure taps. The ΔP data taken with NaK with these taps showed that they were hydrodynamically identical up to velocities of 12 ft/sec. These pressure taps did not become plugged with slurry after thousands of hours of operation. The pressure transducer and read-out system was the same as in the MARK II-P loop. A transformer⁽²⁵⁾ was developed to operate inside the reactor, so that high-current leads used with previous loops could be shortened. A photo of this transformer is shown in Fig. 28. The materials of construction were aluminum, steel, and mica.

The heat exchanger was fabricated from two stainless steel tubes formed in helix and wound together with wire. This form allows the slurry tube to operate 400°C hotter than the coolant tube without violent thermal stressing of the loop. The heat exchanger was tested at low temperatures, and it was found that, with a ΔT of 100°C between tubes, 7 kw of heat could be removed. The cooling system was the same as MARK II-P.

Fig. 26

MARK III-P In-Pile Slurry Loop

Fig. 27

Construction of Pressure
Taps in MARK III-P In-
Pile Slurry Loop

Fig. 28. Radiation Damage-Resistant Transformer

At this time, the slurry program was discontinued and the loop was not placed in the CP-5 reactor. However, depleted UO_2 obtained from Los Alamos Scientific Laboratory⁽²⁶⁾ was used for an out-of-pile experiment. This material was made up of $\sim 10\text{-}\mu$ spherical particles of UO_2 . A microphotograph of material is shown in Fig. 29.

Fig. 29. Photomicrograph of 10-Micron Spherical UO_2 Particles

The loop was cleaned and filled with NaK as in previous experiments. The ΔP data shown in Fig. 30 were taken at 100° C.

Fig. 30. Pressure Drop Data for MARK III-P Slurry Loop at 100° C

After checking out the loop, enough UO_2 to make a 9.3 v/o slurry was added. Typical data at 100° C are shown in Fig. 30. This slurry was circulated for 100 hr. The X-ray photos in Fig. 31 show that the slurry was not completely suspended at the lower flow rates; however, as shown by the ΔP data, most of the material was moving around the loop. The extrapolated ΔP data gave a v/o of 8.7, which corresponded to 32.3 gm not circulating. Figure 32 is an X-ray photo looking down on the helically shaped heat exchanger. It can be seen that the centrifugal force on the particles has caused the UO_2 to flow along the outer edge of the tubing. Photos of the heat exchanger with the 9.3 v/o slurry flowing and settled are shown in Fig. 33A and 33B.

After 100 hr of operation, a second batch of UO_2 was added to make a 18.6 v/o slurry. X-ray photos shown in Fig. 34 shows the 18.6 v/o slurry suspended in the heat exchanger. The denser slurry did not show the separation of solid and vehicle shown by the 9.3 v/o slurry in Fig. 34.

Fig. 31. X-rays of 9.3 v/o Slurry at 100°C Circulating at Various Flow Rates through Heat Exchanger in MARK III-P In-Pile Loop

Fig. 32. X-ray of 9.3 v/o Slurry Flowing through Helical Heat Exchanger

Fig. 33. X-rays of (A) Dynamic and (B) Settled 9.3 v/o Slurry in Heat Exchanger

2.83 fps
T = 50°C

5.66 fps
T = 50°C

Fig. 34. X-rays of Heat Exchanger with 18.6 v/o Slurry Suspended

V. ENGINEERING LOOP TESTS

A series of studies were performed under contract to ANL by the Mine Safety Appliances Corporation on the behavior of UO_2 -NaK slurry in engineering systems. The studies were performed under Contracts 31-109-38-481 and 31-109-eng-38. The purpose of these studies was to determine the characteristics of the UO_2 -NaK slurry at various concentrations and temperatures in conventional piping and component assemblies. In addition, pump-performance characteristics were plotted and matched against the operation of the system with NaK in the absence of UO_2 . To accomplish these objectives, the following operations were performed with the constructed loops:

1. measurement of frictional pressure drop as a function of UO_2 percentage and particle size;
2. measurement of the stability of the slurry in a piping system and the tendency of the UO_2 to separate from the NaK by attachment to the piping walls;
3. methods for changing and maintaining UO_2 concentrations;
4. determination for effective monitoring of slurry density and composition;
5. collection of performance data for electromagnetic pumps in pumping of UO_2 -NaK slurries at various temperatures; and
6. the performance of various types of equipment for the measurement of slurry flowrates.

A. MARK I Engineering Test Loop

1. Loop Description

Fig. 35 is a schematic diagram of the first loop constructed for the studies. As shown, the loop consisted of a main loop and supply loop. The supply loop was composed of the following:

- (1) supply tank for loading of hydrogen-reduced UO_2 and for slurry holdup;
- (2) supply pump and flowmeter; and
- (3) bypass purge pump and associated piping.

The main loop consisted of:

- (1) main pump;
- (2) drain tank;

- (3) test section and density-measuring section;
- (4) venturi and electromagnetic flowmeters; and
- (5) differential pressure (DP) cells connected at various locations, as shown by numbered positions.

A photograph of the partially constructed loop is shown in Fig. 36.

Fig. 36
MARK I Engineering Test Loop
(Partially Constructed)

2. Experimental Procedure

The procedure for operating the loop and collecting the essential data consisted of the following steps.⁽⁶⁾ Initially, the loop was loaded with NaK and operated. Pressure drops were taken across the various positions over the loop. During these runs, the flowmeters were calibrated against each other at temperatures up to 400°F, and a heat balance was determined for the loop. Following these runs, the NaK was drained hot from the loop and the system charged with fresh NaK. Finally, charges of hydrogen-reduced UO_2 were added to the supply tank via the charging valve at the top of the tank to which a mating UO_2 container was affixed. The slurry so formed was now transferred to the main loop and back to the tank as needed.

During the operation of the loop, the volume fraction of the slurry was determined from pressure readings according to the following relationship:

$$x(\rho_p - \rho_{\text{NaK}}) = \frac{(P_3 - P_4) + (P_5 - P_4)}{2z} + (\rho'_{\text{NaK}} - \rho_{\text{NaK}}) ,$$

where

x = slurry volume fraction of UO_2

$P_{3,4,5}$ = pressures measured at points indicated along the loop density-measuring sections

ρ_p = density of UO_2 particle

ρ'_{NaK} = density of NaK in DP pressure lines

ρ_{NaK} = density of NaK

z = difference in elevation of pressure taps.

The determination was based upon the following assumptions:

- (1) The velocity is large enough to achieve a uniform suspension in the riser and downcomer.
- (2) The pressure drop in the riser equals that in the downcomer.
- (3) The pressure taps are reading static pressure only.

Fig. 37. Curve for Determination of True Volume Fraction from Pressure Drop Data

These conditions are not actually realized in practice; therefore, only an apparent volume fraction is actually recorded. If these fractions at various flow velocities are plotted, a curve is obtained as shown in Fig. 37. This curve can be extrapolated to zero velocity to yield the true volume fraction.

The second method used for determining slurry density was from a comparison of the venturi and electromagnetic flowmeter readings.

For the electromagnetic flowmeter,

$$Q = C_1 E \quad , \quad (1)$$

and for the venturi

$$Q = C_2 \sqrt{\Delta P / \rho_f} \quad , \quad (2)$$

where

Q = volume flow rate

E = voltage from flowmeter

ΔP = pressure drop across venturi

ρ_f = density of fluid slurry

C_1, C_2 = flowmeter coefficients.

Combination of Eqs. (1) and (2) yields

$$\rho_f = C_2^2 \Delta P / C_1^2 E^2 \quad . \quad (3)$$

The volume fraction can now be determined from the relationship

$$x = \frac{\rho_f - \rho_{\text{NaK}}}{\rho_p - \rho_{\text{NaK}}} \quad . \quad (4)$$

In addition to the measurements described above, the loop was X-rayed to determine the distribution of UO_2 throughout the system. This operation was performed a number of times during the experimental program.

During the initial calibration runs with NaK only, the loop was operated up to an average temperature of 300°F. The curves for pressure drop versus flowrate for the density-measuring and test sections are shown in Figs. 38 and 39. In addition, the comparison curve for the electromagnetic and venturi flowmeters is shown in Fig. 40. It can be seen that the slope of the line is 1.08, which signifies reasonably good agreement between flowmeters

Following the calibration runs with NaK, the loop was charged with an initial charge of 10 lb of hydrogen-reduced UO_2 . The UO_2 was introduced into the supply tank. X-ray photos taken after the addition of the UO_2 and circulation through the system disclosed that UO_2 was sticking to the walls of the supply tank and large voids were present. Also, the presence of UO_2 plugs in the differential pressure (DP) lines were confirmed with the X-ray photos. Seal pots were added to the system DP lines in an effort to eliminate the problem of plugging. Runs were conducted with the system, and data were collected up to 300°F to determine pressure drops and pump performance. Following these runs, a second addition of 9 lb 12 oz of UO_2 was made. As the runs were continued, difficulty was still encountered with UO_2 plugging the seal pots and DP lines. Trapped gas resulted in the collection of erratic data.

Fig. 38. Pressure Drop as a Function of Flow Rate for Density-Measuring Section

Fig. 39. Pressure Drop as a Function of Flow Rate

Fig. 40. Comparison of EM Flowmeter and Venturi Data

Subsequently, it was decided to attempt wetting of the UO_2 particles by subjecting the slurry to a long-time heat treatment. The slurry was transferred into the supply tank and held at $750\text{--}850^\circ\text{F}$ for a period of 4 hr. The heat treatment proved to be very beneficial since, immediately, several effects were noted. The first was the drop in the NaK level in the supply tank with associated release of gas into the space above the level. Also, the transfer behavior of the slurry proved to be much smoother than previously. Finally, the system experienced fewer difficulties with soft plugs forming in the DP lines. The data for the succeeding runs at 300°F now showed less scatter than the runs before heat treatment.

Fig. 41. Schematic MARK II Engineering Test Loop

B. MARK II Engineering Test Loop

The limitations imposed upon operation of the MARK I test loop which prevented dependable operation at temperatures appreciably above 600°F dictated the necessity for a modified version of the loop to replace the old model. Consequently, the MARK I loop was dismantled and a MARK II loop designed and constructed. The schematic of the MARK II is shown in Fig. 41. In basic principle, the loop is similar to the MARK I model with the addition of several features as follows:

1. Bellows-sealed valves were installed throughout the system to improve integrity and to decrease the possibility of oxygen contamination.
2. A uranium-chip hot trap was installed to permit deoxygenation of the sodium.
3. Horizontal lengths of pipe of various diameters were added to be used in studying the drop-out behavior of the UO_2 .
4. The loop was insulated and designed to permit operation to 1000°F.

Figures 42 and 43 are photographs of the loop in various stages of construction.

Fig. 42. MARK II Engineering Test Loop under Construction

Fig. 43
MARK II Engineering Test Loop Completed (Less Insulation)

When the loop was completed, the system was filled with eutectic NaK and operated successively at 300, 600, and 800°F. Pressure-drop measurements and flowmeter comparisons were made. The results are shown in Figs. 44 through 48. Good correlation was observed between flowmeters.

Fig. 44. Comparison of Venturi and Main Flowmeter Data

Fig. 45. Comparison of Main Flowmeter and Supply Flowmeter Data

Fig. 46

Comparison of Rotameter and Supply
Flowmeter Data

Fig. 47

Test Section Pressure Drop Measurements at 300°F

Fig. 48

Test Section Pressure Drop Measurements at 800°F

In addition, a sample of NaK was removed and its oxygen content determined. The sample analyzed at 0.011 w/o oxygen, a value that indicated saturation at 600°F. Following a hot trapping run of 4 hr with the trap held at 800°F, the oxygen concentration dropped to 0.004 w/o.

With the completion of the preliminary runs, the system was charged with 15.7 lb UO_2 ; the supply tank was heated to 1000°F and held at this temperature for approximately 16 hr to insure wetting of the UO_2 . With this procedure completed, the loop was heated to 300°F and pressure-drop runs were made. After these runs, the loop temperature was raised to 800°F and another series of pressure-drop runs performed.

It was found during these runs that it was not possible to hold the slurry concentration uniform at 800°F. A rapid decrease in slurry concentration was noted as the test progressed. The UO_2 was depositing in the horizontal supply lines. Additional heaters placed upon the upper supply line to keep this line approximately 100°F hotter than the supply loop prevented the drop out in this line.

Determinations of drop-out velocity were also made during this period for the horizontal test sections. The results showed that the slurry of 2 w/o UO_2 at 300°F will start to drop out at velocities of 2.1 to 2.2 ft/sec in horizontal pipes and at 0.21 ft/sec in vertical pipes.

The final runs utilizing this loop were made at 800°F with increasing quantities of UO_2 in the system. Three additions of UO_2 were made (14 lb 7 oz, 16 lb 14 oz, and 28 lb 9 oz) in an effort to achieve a high-concentration slurry. It was not possible to achieve more than a 10 v/o slurry (57 w/o). The UO_2 showed a great tendency to migrate to the cold horizontal pipes, with stagnant NaK pockets, even when these pipes were located higher than the main loop. Initially, large decreases in concentration of UO_2 were noted during the 800°F runs. As before, these changes were reduced considerably by adequately heating the horizontal stagnant NaK lines. At the high concentrations of UO_2 , considerable difficulty was encountered with handling the slurry. Drop out tended to occur very readily; soft plugs and pulsing flow were experienced. At this point, the program was terminated so the loop was cleaned out and dismantled.

C. Results of Engineering Loop Studies

The studies performed with the MSA engineering loops effectively demonstrated the behavior of UO_2 -NaK slurry in conventional engineering equipment.

For the MARK I loop, the pressure drop versus flowrate plots are shown in Figs. 38 and 39, and the comparison curve for the electromagnetic and venturi flowmeters is shown in Fig. 40. During the initial runs with UO_2 in this loop, it was found that good suspension of the UO_2 at 300°F could

be achieved up to 5.6 v/o and that addition and removal of slurry could be effectively accomplished. However, the pressure-drop data show a considerable tendency to scatter if the UO_2 is not adequately wetted by the NaK. This wetting is most readily accomplished by heat treating the slurry at temperatures of 400°C or better for a number of hours.

The pressure-drop flow data for the downstream pressure tap of the test section was correlated to yield slurry viscosity. The following equations were found to represent the data:⁽⁶⁾

$$\left[\frac{\mu_s}{\mu_0}\right]^{0.225} = 1 + 1.2 \times 10^{-5} \times \left[\frac{DV\rho_{\text{NaK}}}{1000\mu_0}\right]^{1.6} - 2.41 \times 10^{-3}x \quad (5)$$

where

$$1.4 < x < 6.0 \quad ;$$

$$10 < V < 27 \text{ ft/sec} \quad ;$$

$$76,000 < \frac{DV\rho_{\text{NaK}}}{\mu_0} < 205,000 \quad ;$$

$$D = 0.465 \text{ in.}$$

and

$$\begin{aligned} \frac{\Delta P}{\rho_s} = \frac{0.0549 V^2 L}{2gD} \left[\frac{DV\rho_s}{1000\mu_0} \right]^{-0.225} \\ + 1.2 \times 10^{-5} \left[\frac{DV\rho_{\text{NaK}}}{1000\mu_0} \right]^{1.6} x - 2.41 \times 10^{-3}x \quad . \end{aligned} \quad (6)$$

In these equations:

μ_s = viscosity of slurry, lb/(ft)(sec)

μ_0 = viscosity of NaK, lb/(ft)(sec)

V = velocity in test section, ft/sec

L = length of pipe, ft

ΔP = measured pressure drop, feet of fluid

ρ_{NaK} = density of NaK

x = volume per cent UO_2 in NaK

ρ_s = density of slurry

Figure 49 is a plot of the observed slurry viscosity against slurry volume fraction and flow velocity. Included in this figure is a plot of the derived equation for the slurry viscosity. It can be seen that the observed

data agree with the equation within $\pm 10\%$. In addition, when Eq. (6) is used to calculate the pressure drop for the test section and the calculated values are plotted against the measured values, the linear plot shown in Fig. 50 results. The values are seen to agree within ± 0.2 psi, showing good agreement for the functions derived.

Fig. 49. Slurry Viscosity as a Function of Volume Fraction and Flow Velocity from ΔP_{9-10}

Fig. 50. Comparison of Observed with Calculated Pressure Drop for ΔP_{9-10}

The necessity for wetting the UO_2 effectively by the NaK was also demonstrated by the improved rheological behavior of the slurry. This was observed in the improved ease with which material transfer could be performed in transfer of slurry between the main loop and storage tank, as well as by the decreased tendency for the formation of plugs in the system.

Several runs were made to demonstrate the drop-out velocity in the horizontal pipe. Figure 51 shows a set of data taken on a 1-in., schedule 80, finned cooler in which drop out occurs at 1.9 to 2.1 ft/sec.

The behavior of the slurry in the vertical supply tank was also studied. At a NaK supply flow of approximately 0.14 ft/sec, instability of

Fig. 51. Dropout of Slurry in Main Loop (MARK I Engineering Test Loop)

flow was very pronounced. X-ray photographs showed that a fluidized bed was building up in the supply tank which produced a variable hydrostatic back pressure in opposition to the flow. Above this region the pressure drop would abruptly decrease.

The effect of slurry upon performance of the electromagnetic pump was also studied. Figure 52 shows the pump correlation for 0, 2, and 6 v/o slurry at 300°F. It can be seen here that the slurry affects the pump performance, but does not introduce any extreme or unexpected variation. The curves for typical pump performance are also shown in the figure for NaK (56 w/o K) at 950°F.

Additionally, the pressure-drop data for the 180° bend were determined. This was done by use of Eq. (6) for the calculation of pressure drop for the 2-in. straight sections and subtraction of the values obtained from the overall measured pressure drops to obtain the ΔP of the bend section. This was necessary since the pressure taps were located 2 in. from the tangent points on the bend. Figure 53 shows the relationship of the pressure drop to the volume fraction of slurry. These data indicated that asymptotic behavior is manifest above approximately 3 v/o UO_2 . The probable cause of this effect is the tendency of the UO_2 particles to settle out in the bend because of centrifugal force. As the buildup of the mass progresses, the channel is restricted, causing an increase in the pressure drop. Eventually, however, the channel restriction forces an increase in the fluid velocity such that further dropout is prevented. After this, the pressure drop does not increase markedly. The plot in Fig. 53 is made with the best curve drawn to fit the data.

In the MARK II engineering test loop, two calculations of pressure drop were made with the use of the friction

Fig. 52. Pump Correlation Curves for 0, 2, and 6 v/o Slurry at 300°F

Fig. 53. Pressure Drop as a Function of Slurry Volume Fraction in Bend of Loop

factor for commercial pipe. The results are shown in Table V, and a comparison is made between observed and computed pressure drops. The pressure drops were calculated by means of the Richter correlation. The equivalent length of a bend (in pipe diameters) is given by

$$Le = 0.202 \times \alpha^{1.1} (Re)^{0.032}, \quad (7)$$

where

Le = equivalent length - pipe diameter

x = factor in Richter equation

α = bend angle

Re = Reynolds number.

Table V

COMPARISON OF OBSERVED AND CALCULATED PRESSURE DROPS

Flow (gpm)	Temperature (°F)	ΔP_{7-6} (feet of fluid)	
		Observed	Calculated
15.0	800	3.35	3.34
13.5	300	3.1	3.11

The equivalent length of the bends was added to the straight lengths of pipe existing between pressure taps 7 and 6. Then the pressure drop was calculated to yield the data in Table V.

The drop-out runs showed that the slurry starts to drop out in horizontal pipes at velocities of 2.1 to 2.2 ft/sec, and this seems to be independent of pipe diameter. The vertical drop-out velocity was equal to 0.21 ft/sec. The drop-out velocity is defined as the minimum velocity which will maintain slurry concentrations. The data collected in the MARK II slurry loop for UO_2 concentration of 2 v/o at 300°F are shown in Table VI.

At an 800°F loop temperature, the UO_2 was observed to deposit out in the cooler horizontal supply lines. When electrical heaters were installed to maintain the supply lines 100°F higher than the main loop, the concentration was not observed to decrease. During the loop heating, the

concentration was observed to remain constant until the loop temperature reached 650-700°F. Above this temperature range, the concentration was seen to drop until the supply temperature was raised above that of the main loop.

Table VI

DROP-OUT VELOCITIES, UO_2 -NaK SLURRY IN
MSA MARK II ENGINEERING LOOP
(2 v/o Slurry at 300°F)

Pipe		Velocity (ft/sec)
Size (in.)	Position	
$\frac{3}{4}$	Horizontal	2.27
1	Horizontal	2.1
$1\frac{1}{2}$	Horizontal	2.23
2	Horizontal	2.16
3	Vertical	0.21

Fig. 54. Pressure Drop of
3 v/o UO_2 -NaK Slurry
at 800°F (MARK II
Engineering Test Loop)

At 800°F, the slurry pressure drops were determined, and these results are plotted in Fig. 54. These data are for a 3 v/o slurry. Table VII gives a comparison of the pressure drop at 800°F between pure NaK and UO_2 -NaK slurry. It can be seen that the pressure drop in feet of fluid is ~10% higher than for the pure NaK case.

The findings with the MARK II loop indicated that a 10 v/o slurry can be circulated. However, at higher concentrations, the UO_2 is wedged against the walls, and the pressure drop becomes quite high. If side piping is present, the UO_2 will be driven into these lines, dropping the concentration to 10 v/o. In addition, at high concentrations, the slurry would, at times, exhibit fluctuating flow, indicating the formation and passage of "plugs." In

vertical sections, a check valve type of behavior would be observed. The UO_2 would plug at the inlet point and would permit only a small downward

flow of NaK, while it affected the upward flow only slightly. The type of behavior at these high concentrations places the concentration limitations upon the slurry at 10% unless pumping of a paste is considered, or a special configuration is designed as described in the in-pile loop section.

Table VII

COMPARISON OF NaK AND SLURRY PRESSURE DROP

Flow (gpm)	NaK (ft of fluid)		Slurry (ft of fluid)	
	ΔP_{7-6}	ΔP_{8-7} ΔP_{6-5}	ΔP_{7-6}	ΔP_{8-7} ΔP_{6-5}
8	3.02	1.46	3.3	1.45
20	18.1	8.45	19.0	9.5

VI. CONCLUSION

The studies which were conducted during this program have effectively demonstrated the behavior of the $\text{UO}_2\text{-NaK}$ slurry system both in- and out-of-pile. It is clear that one cannot expect to maintain slurry concentrations above 10 v/o in a system composed of conventional hydraulic components. However, higher concentrations can be handled in systems tailored to eliminate the problems encountered with slurry in the normal assemblages.

The in-pile behavior of the slurry was shown to be as expected. Under proper conditions one can utilize the attractive characteristics of this type of fuel, i.e., high heat transfer rates, uniform burnup, freedom from destructive irradiation damage, etc. However, the number of tests conducted and the burnups achieved were not sufficiently numerous to study satisfactorily the entire spectrum of possibilities. In addition, slurries of other combinations of liquid metal and fissionable material carried in suspension have not been investigated for possible superior features which are lacking in the present combination studied. It appears, however, that with the present state of knowledge concerning the $\text{UO}_2\text{-NaK}$ slurry that a limited potential does present itself in the utilization of this material. One reactor design using this material in a helically positioned core has been patented.⁽²⁷⁾ Another application of such a fueled system which may be considered is in satellite power units designed to operate in zero gravity where the drop-out problem would not appear and where heat transfer conditions become considerably different than normally encountered in the presence of gravity.

In conclusion, it is urged that the attractiveness of the slurries should be considered and attempts to apply the material should be continued.

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APPENDIX A*

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