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**Waste Sludge Resuspension and
Transfer—Development Program**

H. O. Weeren
T. S. Mackey

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UNION CARBIDE CORPORATION
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DEPARTMENT OF ENERGY

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NUCLEAR FUEL AND WASTE PROGRAMS
Gunite Tank Sludge Removal/Disposal
(Activity No. AR 05 10 05 K; 189 No. ONL-WN03)

WASTE SLUDGE RESUSPENSION AND TRANSFER — DEVELOPMENT PROGRAM

H. O. Weeren
T. S. Mackey*

*Operations Division.

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*WASTE SLUDGE RESUSPENSION AND TRANSFER —
DEVELOPMENT PROGRAM*

H. O. Weeren
T. S. Mackey*

HIGHLIGHTS

The six Gunitite waste tanks at Oak Ridge National Laboratory (ORNL) contain about 400,000 gal of sludge that has precipitated from solution and settled during the 35 years these tanks have been in service. Eventual decommissioning of the tanks has been proposed. The first part of this program is to resuspend the accumulated sludge, to transfer it to new storage tanks in Melton Valley, and to dispose of it by the shale-fracturing process.

On the basis of preliminary information, a tentative operational concept was adopted. The sludge in each tank would be resuspended by hydraulic sluicing and pumped from the tank. This resuspended sludge would be treated as necessary to keep the particles in suspension and would be pumped to the new waste-storage tanks. Subsequently the sludge would be pumped from the tanks, combined with a cement-base mix, and disposed of by the shale-fracturing facility. Verification of the feasibility of this concept required development effort on characterization of the sludge and development of techniques for resuspending the sludge and for keeping it in suspension. These development efforts are described in this report.

Sections of the report describe both the known properties of the sludge and the tests of grinding methods investigated, discuss tests of various suspenders, describe tests with cement-base mixes, summarize hot-cell tests on actual sludge samples, and describe tests that were made at a mockup of a Gunitite tank installation.

On the basis of the tests made, we concluded that reslurrying and resuspension of the sludge is quite feasible and that the suspensions can be made compatible with cement mixes. A modification of the original operating concept was made, in which a batch of a bentonite suspension will be prepared in an empty Gunitite tank. This suspension will be pumped to the sluicing jet in the tank being cleaned, and the resuspended sludge will be pumped back into the tank containing the suspension. Circulation will be continued until the concentration of resuspended sludge reaches the desired value.

*Operations Division.

1. INTRODUCTION

The six Gunitite waste tanks at ORNL contain about 400,000 gal of sludge that has precipitated from solution and settled during the 35 years these tanks have been in service. As a first step in a program for the eventual decommissioning of these tanks, it is proposed that this sludge be resuspended; transferred to new, doubly contained, stainless steel tanks in Melton Valley; and disposed of by the hydrofracture process. This report describes the development work that has been done to characterize the sludge, to devise a technique for removal of the sludge from the existing tanks, to select suspenders for the sludge, and to determine compatibility of the suspended sludge with the shale-fracturing grout mixes.

Each of the six waste-storage tanks in the ORNL tank farm is 50 ft in diameter and has a vertical height of 12 ft. The dome of each tank rises an additional 6 ft and is covered with several feet of earth. A sketch of a typical tank is shown in Fig. 1. Each tank has a capacity of about 170,000 gal. The tanks were built in 1943 of steel-reinforced Gunitite (a trade name for a mix of cement, sand, and water sprayed from a cement gun). The six tanks are arranged in a 60-ft center-to-center square matrix and are numbered consecutively, W-5 to W-10.

Preliminary measurements of the depth of the sludge interface indicated that the volume of sludge in each tank varied from 10,000 gal (W-10) to 136,000 gal (W-5). Samples of these sludges contained 30 to 60 wt % solids. A large fraction (50 to 90%) of these solids consisted of very small particles ($<10\text{ }\mu\text{m}$). The remaining fraction consisted of agglomerates of these particles which were as large as $1/8\text{ in.}$ diam and were quite friable. On the basis of this preliminary information, we adopted a tentative operational concept. The sludge in each tank would be resuspended with a water jet and pumped from the tank. The resuspended sludge would be pumped through the waste-transfer pipeline (2 in. diam) to the new waste-storage tanks in Melton Valley (some grinding of the large sludge particles would probably be required before the sludge could be safely pumped). Subsequently, the sludge would be pumped from the new waste-storage tanks, mixed with cement, and disposed of by the shale-fracturing

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Fig. 1. Cross section of waste tank.

facility. The firming of this tentative concept required considerable research and development effort to characterize more completely the sludge, to develop techniques for suspending the sludge and for keeping it in suspension, to make large-scale sluicing and pumping tests, to test the applicability of various instruments, to evaluate grinding methods, and to determine compatibility of the sludge with the shale-fracturing grout mixes.

2. SLUDGE CHARACTERIZATION

Each of the waste tanks has a very limited number of openings; most of the tanks have only one opening available through which a sample of the sludge can be taken. The properties of the sludge could vary appreciably from point to point within a tank, but a large number of samples would be required to characterize even approximately the sludge contents of a single tank. The usefulness of such a program was therefore deemed questionable. Instead, at least one sample was obtained from each tank; five samples were obtained at different depths from W-5, W-6, and W-8, the tanks containing the greatest volume of sludge. In some cases, multiple samples were obtained. These samples were analyzed for physical properties [density, weight fraction solids, percent fines ($<10\ \mu\text{m}$)], radiochemical composition, and chemical composition.

2.1 Sludge Volume

The volume of sludge in each tank was obtained from a measurement of the sludge level. Both sludge "volume" and "level" are somewhat nebulous concepts; the interface between the sludge and the supernatant solution is almost certainly a zone rather than a sharp separation. The sludge volumes given in this report are therefore only approximations. The reported values have varied appreciably over the years; whether or not this variation results from an actual change or merely represents a variation in measuring technique is unknown. These reported volumes are given in Table 1.

Table 1. Reported sludge volumes in Gunitite tanks (gal)

	Tank number					
	W-5	W-6	W-7	W-8	W-9	W-10
1964	60,000	86,000	30,000	86,000	12,000	10,000
1970	136,000	45,000	22,300	132,000	18,700	9,600
1975	139,500	58,700		98,400		

2.2 Sampling Devices

Three types of devices have been used to obtain samples of the sludge in the Gunitite tanks. The first device was used in 1972 and consisted of a short section of 3/4-in. pipe having one side cut open and flared to form a scoop. This device was lowered into the tank at the end of several jointed lengths of pipe until the bottom of the tank was reached. The sampler probe was then withdrawn about 2 in., rotated to scoop sludge into the sampler head, and withdrawn from the tank. A sketch of the sampler head is shown in Fig. 2.

In 1974 and 1975 samples of the sludge in tanks W-5, W-6, and W-8 at several precise depths were needed for estimating the variation, with depth, in concentration of sludge components. The sampler previously used would not keep separate the samples taken at different levels; we therefore designed and built a new sampler. A sketch of this sampler head is shown in Fig. 3. The sampler head contains five sample holes, each 6.5 in. long and 3/8 in. in diameter. An indexing mechanism allowed each hole to be opened as the sampler head reached a precise depth in the sludge.

In 1977 large sludge samples for grinding and settling tests were obtained from tanks W-5, W-6, and W-8 by use of a soil sampler. This soil sampler consists of a hollow tube 1-3/8 in. ID and 18 in. long attached to the end of a long shaft. A retainer assembly at the bottom of the tube prevented the sample from flowing out of the head as it was withdrawn from the tank. The sample obtained with this sampler was a "composite" sample and was not characteristic of any particular depth in the tank.

Fig. 2. Head detail of first sludge sampler.

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Fig. 3. Head detail of second sludge sampler.

2.3 Physical Properties

The measured physical properties of the sludge samples include sludge density, weight fraction solids, fraction of solids that settles rapidly in water, radiation dose rate, and friability. Not all of these measurements were made on all samples.

2.3.1 Sludge density

The sludge density was measured by adding a known weight of water to a known weight of sludge in a graduated cylinder, stirring well, measuring the volume of the diluted sludge, and calculating the density of the original sludge from these results. The results obtained from these density determinations are shown in Table 2.

Table 2. Sludge density (g/cm³)

Level ^a		W-5	W-6	W-7	W-8	W-9	W-10
1973		1.61	1.5		1.21		
1974		1.28	1.33	1.0	1.08	1.25	1.31
1975	1	1.17	1.30		2.37		
	2	1.30	1.42		1.28		
	3	1.18	1.51		1.32		
	4	1.50	1.61		1.28		
	5	1.76	1.38		1.22		
1976	1	1.58	1.28		1.96		
	2	1.79	0.76		1.51		
	3	1.69	1.15		2.46		
	4	2.23	1.85		2.20		
	5	1.67	1.42		1.57		
1977		3.8	1.92		2.30		

^aIn tank W-5, level 1 is 7.7 ft from bottom, level 2 is 6.2 ft from bottom, level 3 is 5.2 ft from bottom, level 4 is 3.2 ft from bottom, and level 5 is 1.7 ft from bottom; in tank W-6, level 1 is 2.8 ft from bottom, level 2 is 2.5 ft from bottom, level 3 is 1.8 ft from bottom, level 4 is 1.5 ft from bottom, and level 5 is 0.8 ft from bottom; in tank W-8, level 1 is 5.7 ft from bottom, level 2 is 4.7 ft from bottom, level 3 is 3.7 ft from bottom, level 4 is 2.7 ft from bottom, and level 5 is 1.7 ft from bottom.

The values obtained show much variation, even for samples taken at the same level in the same tank. Also, some values are improbably low (1.0 or less) and some values are almost impossibly high (3.8 for tank W-5 in 1977). Finally, many of the values cannot be reconciled for the fraction of solids in the sludge (Sect. 2.3.2) and any reasonable particle density. For the sludge density to be greater than 2.0, for example, the particle density must be greater than 6.0 or the fraction solids must be greater than 0.6. A probability is that the reported sludge density values are subject to appreciable error.

2.3.2 Sludge solids concentration

The weight fraction of the sludge, consisting of insoluble particles, was measured by washing a known weight of sludge several times to remove soluble components, drying, and weighing the residual sludge. The results obtained are shown in Table 3. The most probable cause for error in these results would be the loss of a small fraction of small-diameter particles not settled after washing, which would make the reported results somewhat low. The results in Table 3 vary considerably from point to point but seem to show a solids concentration ranging from 25% near the top of the sludge to 55% near the bottom in tank W-5, a similar range of concentration in tank W-6, and a fairly uniform 35% concentration in tank W-8.

2.3.3 Particle size distribution

The first sludge samples showed that the sludge contained a large fraction of very small particles that settled slowly in water, which led us to the conclusion that these particles were less than 10 μm in diameter. Microphotographs of the fines showed particle diameters of less than 2 μm . We estimated the fraction of the sludge solids of less than about 10 μm in diameter for some of the samples by shaking the sludge sample with excess water, allowing it to settle for 2 min, decanting the unsettled particles, and weighing the remaining particles. Calculation of the rate of unhindered settling in water of particles having a density of 4.0 indicated that the diameter of the unsettled particles would be about 15 μm .

An approximate particle size distribution was determined for the large sludge samples obtained from tanks W-5, W-6, and W-8 in 1977. In these

Table 3. Concentration of solids in sludge (wt %)

	Level	W-5	W-6	W-7	W-8	W-9	W-10
1974		55.0	50.0	21.0	23.0	23.0	44.0
1975	1	25.8	48.3		16.8		
	2	12.1	57.8		49.0		
	3		49.7		30.1		
	4	42.7	37.6		37.3		
	5	59.6	51.8		27.5		
1976	1	26.0	24.2		33.3		
	2	22.8	25.3		30.5		
	3	20.3	29.2		38.2		
	4	20.2	25.4		30.1		
	5	46.4	37.3		36.9		
1977			29-43 ^a		34-37 ^a		

^aRange of three values from 18-in. core sample.

tests the particle distribution in the size range of 1 to 100 μm was obtained. The results indicate that about 10% of the particles in each tank were smaller than 15 μm . These results are quite different from results obtained earlier, which indicated more than 50% fines. The 1977 tests were made with samples that had been dried prior to use; the earlier tests were made with samples that had not been dried. The drying step probably caused some particle agglomeration and biased the results, which are given in Table 4.

2.3.4 Physical appearance of slurry particles

Microphotographs were taken of sludge particles from tanks W-5, W-6, and W-8, which show a considerable variability in the size and shape of the particles in different samples. In general, however, the sludge is composed of discrete particles, with the larger particles being agglomerations of the smaller particles. Figures 4 and 5 are pictures of small-size and large-size fractions of particles from tank W-6. In Fig. 4, the largest particle is about 2 μm long. In Fig. 5, the largest particles in the center of the picture are about 250 μm in diameter.

Fig. 4. Sludge particles, high magnification.

ORNL PHOTO NO. 0544-80

Fig. 5. Sludge particles, medium magnification.

Table 4. Particle size distribution in sludge

		W-5	W-6	W-7	W-8	W-9	W-10
1972	Wt % solids <15 μ m	25	45		100		
1974	Wt % solids <15 μ m	59	65	73	82	61	7
1977	Wt % solids >60 μ m	82	91		86		
	Wt % solids >40 μ m	87	92		88		
	Wt % solids >10 μ m	91	95		90		
	Wt % solids <10 μ m	9	5		10		

2.3.5 Radiation dose rates from sludge samples

Crude radiation dose rates were made of the samples taken from each tank in 1974. In each case a weighed sample was read at a distance of 1 in. with a portable radiation detector (cutie pie), following which the sample was diluted with water and read again. The results were:

Tank W-5: 4.6-g sample read 500 mR at ~1 in.;
when diluted to 22 ml, read 100 mR

Tank W-6: 1.6-g sample read 700 mR; when
diluted to 7.6 ml, read 100 mR

Tank W-7: 2.8-g sample read 400 mR; when
diluted to 14 ml, read 200 mR

Tank W-8: 1.3-g sample read 700 mR; when
diluted to 6.4 ml, read 300 mR

Tank W-9: 3-g sample read 200 mR; when
diluted to 14.4 ml, read 80 mR

Tank W-10: 2.1-g sample read 300 mR; when
diluted to 10 ml, read 250 mR

2.3.6 Sludge particle friability

Observation of the 1972 sludge samples revealed that the larger particles could be crushed quite easily into smaller particles. Early settling tests had indicated that small sludge particles could be kept in suspension much more easily than could the larger particles; a particle crushing or

grinding therefore seemed likely to be a probable process step. Because of the problems of crushing and grinding actual sludge particles, a substitute material was sought — one that would approximate the difficulty of grinding of the sludge particles but would be nonradioactive.

A friability test was made on sludge particles from each of the Gunitite tanks. A part of each 1974 sample was suspended in excess water, and the particles having diameters larger than 20 μm separated by settling. The settled fraction was dried, a weighed amount was placed on a 60-mesh screen and shaken, and the fraction that passed through the screen was noted. Several steel balls were added to the coarse fraction that remained on the screen, the screen was shaken, and the weight of material that passed through the screen after 5, 10, and 15 min was noted. Table 5 gives these results. Similar results with two grades of Chromosorb (a diatomaceous earth packing material) are given for comparison. On the basis of these results, we selected Chromosorb as a sludge substitute for subsequent grinding tests.

Table 5. Friability test results
(cumulative percentage through 60-mesh screen)

	5 min	10 min	15 min
W-5	26.7	83.4	89.6
W-6	18.2	95.9	98.6
W-7	25.1	82.1	95.8
W-8	28.7	99.0	100.0
W-9	7.8	98.8	100.0
W-10	15.3	62.5	98.8
Chromosorb W	19.4	53.4	75.8
Chromosorb P	12.0	27.6	46.0

2.4 Radiochemical Analysis

Samples taken from different levels in tanks W-5, W-6, and W-8 in 1975 and 1976 were assayed for radionuclides. The reported concentrations of these radionuclides were multiplied by the reported sludge density, by the reported solids concentration, and by the sludge volume in the sampled

zone to estimate the total quantity of each radionuclide in the sample zone. The quantities of each radionuclide in each sample zone were then summed to estimate the tank load. These values are given in Table 6 for both sets of samples. The quantities of each radionuclide in each zone are given in the Appendix.

The values given in Table 6 are a summation of the product of three factors — sludge density, weight fraction solids, and reported radionuclide concentration. As indicated in Sects. 2.3.1 and 2.3.2, the reported values for sludge density and weight fraction solids show considerable variation and some improbability. The reported values for radionuclide concentrations also show considerable variation between samples. It is not surprising, therefore, that the products of these numbers show anomalies. An extreme example is illustrated by the ^{239}Pu analysis at the 3.2-ft level in tank W-5. The calculated quantity of this radionuclide in this tank volume increment was 748 Ci, based on the 1975 results, and 19 Ci, based on the 1976 samples. The sludge density in the two samples varied by a factor of 1.5, the percent solids varied by a factor of 2.1, the concentration of alpha emitters by a factor of 3.0, and the fraction of ^{239}Pu in the alpha emitters by a factor of 70. Other radionuclides exhibit similar variation. Obviously, the sludge composition is not uniform.

2.5 Chemical Analysis

Fractions of the sludge samples taken in 1975 were dried and analyzed by x-ray diffraction. The major reported constituents were Al, S, Cl, Ca, Fe, Pb, Th, and U (elements smaller than Na cannot be detected by this technique).

A few x-ray diffraction patterns were recognized, which were characteristic of $\text{Na}_2\text{U}_2.5\text{O}_{8.5}$ and $\text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O}$ from a sample from tank W-5 and of $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ and Fe_2O_3 from two samples from tank W-6.

The results from x-ray diffraction measurements were correlated with previously available data on sample density, fraction solids, and weight fractions uranium and thorium to yield the approximate overall composition given in Table 7. The reported data for each sample are given in the Appendix.

Table 6. Radiochemical content of sludge (Ci)

Radionuclide	Tank	1975	1976
^{90}Sr	W-5	3.9×10^5	8.23×10^5
	W-6	8.8×10^4	1.99×10^5
	W-8	1.33×10^6	4.1×10^5
^{60}Co	W-5	4.0×10^3	6.9×10^3
	W-6	176	2.4×10^3
	W-8	3.3×10^3	1.4×10^5
^{106}Ru	W-5	2.3×10^3	7.8×10^3
	W-6	497	4.8×10^3
	W-8	2.2×10^3	1.5×10^3
^{137}Cs	W-5	4.5×10^3	3.9×10^3
	W-6	9.5×10^3	4.9×10^3
	W-8	2.4×10^4	5.3×10^4
^{144}Ce	W-5	1.4×10^3	1.5×10^3
	W-6		3.9×10^3
	W-8		946
^{154}Eu	W-5	4.0×10^3	9.1×10^4
	W-6	104	413
	W-8	1.0×10^4	1.1×10^3
^{152}Eu	W-5	1.1×10^3	2.1×10^3
	W-6	61	
^{239}Pu	W-5	1.0×10^3	1.47×10^3
	W-6	237	
	W-8	231	236
^{244}Cm	W-5	5.5×10^3	1.27×10^4
	W-6	523	6.2×10^3
	W-8	103	3.0×10^3

Table 7. Composition of solids in Gunite tanks (wt %)

Element	Tank		
	W-5	W-6	W-8
Uranium	22.9	1.5	3.7
Thorium	6.4	0.8	14.2
Aluminum	1.0	10.3	5.0
Calcium	6.5	2.9	10.3
Iron	1.0	18.5	2.9
Lead	2.2	4.0	<i>a</i>
Sulfur	0.6	<i>a</i>	3.7
Phosphorus	1.7	<i>a</i>	<i>a</i>
Chlorine	1.0	2.7	0.7

^aSmall.

3. TEST LOOP OPERATION

The operational concept for emptying the Gunite tanks is based on resuspending sludge particles and pumping the resulting slurry through the waste-transfer pipeline to the new intermediate-level waste (ILW) storage tanks. Preliminary information was needed on the flow characteristics of slurries that were similar to the resuspended sludge to establish the validity of the concept and to recognize obvious potential problems. To obtain this information and to test the applicability of several types of instruments to slurry flow, we built a test loop and operated it with several different materials as substitute slurries.

3.1 Slurry Properties

The theoretical treatment of the flow of slurries in pipes divides these slurries into two types having quite different characteristics — homogeneous (nonsettling) slurries and heterogeneous (settling) slurries. Data obtained from sludge samples indicated that a large fraction of the sludge particles would be smaller than 10 μm and that the remaining fraction

could be broken into small particles with little difficulty. Particles of this diameter either would form a nonsettling slurry or could be made to do so by the addition of a small amount of suspender, such as bentonite. Because a nonsettling slurry would be much safer to pump through the existing pipeline to the new ILW tanks, we assumed that this type of slurry would be generated during the resuspending operation (by treatment, if necessary) and that the experimental work should be done with nonsettling slurries.

Microphotographs of "fine" sludge particles show that these particles are quite discrete with a particle diameter of 2 μm or less. Settling tests with these fines indicated that the average particle diameter was 10 μm or less. The close agreement between these separate measurements of particle size indicates that very little particle interaction occurs. The agreement also suggests that tests run with other discrete and non-reactive particles would give results similar to those from tests with actual sludge slurries and could therefore be extrapolated to these slurries without serious error. For the descriptions in this section, slurries of pottery clay, fly ash, and black iron oxide (Fe_3O_4) were tested. A few tests were made, using mixed oxides and a suspender. All of these slurries will settle after standing (as will the sludge slurries). In all cases, however, this settling rate is low enough so that, under flow conditions, these slurries are nonsettling slurries in which the particles and transporting liquid interact physically to such an extent that the resulting fluid has properties distinctly different from those of the carrier liquid.

3.2 Instruments

Many instruments that work well in fluid systems work poorly or not at all in slurry systems. Two devices that supposedly would work well in slurries were selected for trial in the test loop.

An elbow meter consists of a pipe bend with pressure taps inside and outside the bend. The differential pressure between the two taps is supposedly proportional to the square of the velocity of the flowing fluid. This type of meter is reputedly insensitive to changes in the specific gravity and concentration of the solids present.

A vertical counterflow meter consists of two vertical sections of pipe, in which the slurry flows upward in one and downward in the other. The differential pressure between two points on each section is measured and related to the slurry velocity and the slurry density. A sketch of this device is shown in Fig. 6. The slurry velocity is proportional to the square root of the sum of the two differential pressures, and the slurry density is proportional to the difference of the two differential pressures.

An ultrasonic flow meter (Scarpa Laboratories) was tested briefly. This meter is a clamp-on type that senses the high-frequency component of pumping noise. This component would presumably be proportional to slurry velocity.

3.3 Test Loop

A sketch of the test loop is shown in Fig. 7. The loop consists of an agitated slurry tank, a pump, a counterflow meter, an elbow meter, a "trap," and a head tank. The head tank was used to determine the rate of slurry flow. The trap was a transparent section of line with a built-in low point; it was installed to observe the settling characteristics of the slurry during pumping and after shutdown. The loop was made of 0.5-in.-diam, 0.035-in.-wall tubing. The manometers used to measure pressure drops were filled with fluid having a density of 2.95 g/cm^3 . A small flow of purge water was provided to each leg of each manometer to keep the pressure taps flushed clear of slurry particles.

The loop was tested with several slurries — a slurry of 14 wt % fly ash, a slurry of 20 wt % pottery clay, a slurry of 30 wt % pottery clay, a slurry of 10 wt % black iron oxide (Fe_3O_4), a slurry of 15 wt % black iron oxide, a slurry of 15 wt % Fe_3O_4 plus 1 wt % bentonite, and a mixed slurry of 5 wt % Fe_3O_4 , 5 wt % CaCO_3 , and 3 wt % bentonite. All of the slurries pumped easily. No settling in the trap was observed during loop operation; those particles that settled during loop shutdown were immediately resuspended when the pump was restarted. Some difficulty was experienced in keeping the Fe_3O_4 slurry in suspension in the feed tank; the dense particles tended to settle in the tank, and additional agitation had to be provided.

$$h_1 = h_f + LC(S-1)$$

$$h_2 = h_f - LC(S-1)$$

h_f = FRICTION HEAD, ft

L = LENGTH BETWEEN TAPS, ft

C = VOLUME FRACTION SOLIDS

S = SPECIFIC GRAVITY OF SOLIDS

AS DENSITY METER

$$h_1 - h_2 = 2LC(S-1)$$

$$C = \frac{h_1 - h_2}{2L(S-1)}$$

AS VELOCITY METER

$$h_1 + h_2 = 2h_f$$

$$h_f = \frac{2fLV^2}{g_c D}$$

$$V = K[h_1 + h_2]^{1/2}$$

$$\text{WHERE } K = \left[\frac{g_c D}{4fL} \right]^{1/2}$$

Fig. 6. Vertical counterflow meter.

Fig. 7. Slurry test loop arrangement.

A typical run with a single type of slurry consisted of operation at a particular slurry velocity for a time sufficiently long for manometer readings to stabilize. Readings were taken and the flow rate was determined by noting the time required to accumulate 0.5 gal of slurry in the head tank. The head tank was allowed to drain, and a different slurry velocity was selected for the next run. Slurry velocities between 1.5 and 4.5 ft/sec were generally attainable. At velocities of less than 1.5 ft/sec, flow was generally unstable, but velocities greater than 4.5 ft/sec could not be attained with the existing equipment. The slurry density was measured in the feed tank by a hydrometer or by a bubbler system.

3.4 Calculation of Results

Data from a typical run are given in Table 8. These results were obtained with a 15 wt % slurry of Fe_3O_4 .

Table 8. Test loop results

Data point	Time for 0.5/gal (sec)	Density (g/cm ³)	Elbow meter (cm oil)	Counterflow meter		Sonic meter
				h_1 (cm oil)	h_2 (cm oil)	
1	16.1	1.0417 ↓	6.0	30.6	11.6	5, H scale ^a
2	16.0		5.2	29.9	11.1	20, H scale
3	14.5		4.8	30.7	11.8	5, H scale
4	23.0		4.8	23.5	3.2	0, H scale
5	25.0		3.2	20.8	0.6	5, H scale
6	25.2		3.0	20.5	0.4	0, H scale
7	81.0		2.1	13.4	-9.7	0, H scale
8	43.5		2.1	16.2	-6.6	5, H scale
9	57.0		1.6	15.2	-7.0	0, H scale

^aH scale = high scale.

The measured time for 0.5 gal is converted to a velocity by dividing by the known tube area. The elbow meter readings are converted to inches of water; results are plotted in Fig. 8.

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Fig. 8. Pressure differential measurements in elbow meter.

The fraction, by volume, of solids C in the slurry is determined from the counterflow meter readings by subtracting h_2 from h_1 , converting this difference to feet of water, and dividing this value by $2L(S - 1)$ (where L is the length between taps in feet, S is specific gravity of solids, h_f is friction head in feet, $h_1 = h_f + LC(S - 1)$, and $h_2 = h_f - LC(S - 1)$). For Fe_3O_4 , $S = 5.1$; for this test loop, $L = 5$ ft. The density of the slurry is then $1 + C(S - 1)$, and the weight fraction of solids in the slurry is $SC/[1 + C(S - 1)]$. The calculated values for these quantities are shown in Table 9.

Table 9. Slurry density measurements

Data point	Volume fraction, C	Slurry density (g/cm ³)	Weight fraction
1	0.0296	1.1214	0.1346
2	0.0293	1.1201	0.1334
3	0.0295	1.1210	0.1342
4	0.0317	1.1300	0.1431
5	0.0315	1.1292	0.1422
6	0.0314	1.1287	0.1419
7	0.0360	1.1476	0.1600
8	0.0356	1.1460	0.1584
9	0.0346	1.1419	0.1546

The slurry flow rate is determined from the counterflow meter readings by a relatively simple iterative calculation. First, the readings h_1 and h_2 are converted to feet of water, added, and the square root is taken. Second, a velocity v is assumed, a Reynolds number Re is calculated (with the slurry viscosity taken to be that of water), and a Fanning friction factor f is determined. Third, the value of $(g_c D/4fL)^{1/2}$ is calculated. Fourth, the velocity is determined as the product of $(h_1 + h_2)^{1/2}$ and $(g_c D/4fL)^{1/2}$. If the calculated velocity is sufficiently different from the velocity assumed in step 2 to have an appreciable effect on f , a different velocity is assumed and steps 2, 3, and 4 are repeated. Because f is relatively insensitive to velocity, an iteration is seldom necessary.

The results obtained from the sample run are plotted in Fig. 9 as indicated velocity (counterflow meter) versus measured velocity. The agreement indicates a constant bias of 20%; the tube diameter was possibly somewhat smaller than assumed.

3.5 Test Loop Results

Data were obtained and calculated from test runs with several different slurries. In general, we observed that all of the slurries were pumpable. All slurries settled in the trap section when the loop was shut off, but the settled particles promptly resuspended when flow was resumed. The iron oxide slurries tended to settle in the feed tank despite rather vigorous agitation of the tank; density measurements of the slurry in the feed tank did not agree with either a mass balance on the loop or the counterflow meter measurements. The sonic meter results were not meaningful. Results from the elbow meter were somewhat erratic during any particular run and were also dependent on the properties or concentration of the slurry being pumped. Further work with the elbow meter did not seem to be justified.

The results obtained with the counterflow meter were encouraging. The measurements of slurry density were consistent and believable. The measurements of pressure drop were generally consistent, and, except for a constant bias, the calculated velocity correlated well with the measured volumetric flow rate. The meter gave consistent results with every slurry that was tested, with one exception. Two runs were made with a slurry of 5 wt % Fe_3O_4 , 5 wt % CaCO_3 , and 3 wt % bentonite. The rheological properties were measured with a Fann viscometer; the slurry was found to be a non-Newtonian power-law fluid with $n' = 0.274$ and $K' = 0.043$. The procedure followed in calculating an indicated velocity is the same as that used for Newtonian fluids, except that the formula used to calculate the Reynolds number is:

$$\text{Re} = [1.86(V)^{2-n'} \rho / K'(96/D)^{n'}],$$

where V is in ft/sec, ρ is in lb/gal, and D is in inches. The indicated velocities calculated for these runs were about 67% of the measured velocities and about 55% of the indicated velocities expected from previous runs.

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Fig. 9. Comparison of indicated velocity in counterflow meter with measured velocity.

An analysis of these results indicated that the results would be consistent if the rheological properties were $n' = 0.35$ and $K' = 0.027$. A reexamination of the Fann viscometer readings suggested that either set of numbers could be derived from the data (the rheological properties of slurries are difficult to measure precisely). The counterflow meter readings are obviously quite sensitive to rheological properties, and these properties must be known precisely if the counterflow meter is to be used to determine slurry velocity. On the other hand, if the slurry velocity is determined by other means, the readings of the counterflow meter can be used to determine the rheological properties of the slurry.

4. GRINDING TESTS

About 50% of the sludge particles in samples taken from the Gunit tanks were less than 10 μm in diameter and settled very slowly in water. The larger sludge particles were observed to be predominately agglomerates of the smaller particles. These observations suggested the operating concept of breaking the sludge agglomerates into their constituent particles. The resulting slurry of fine particles could therefore be pumped through the existing pipeline to the shale-fracturing facility with lessened risk of particles settling out of the slurry and possibly plugging the line.

Tests of the relative friability of sludge agglomerates and two types of Chromosorb (a diatomaceous earth packing material) showed that the Chromosorb was less friable (Sect. 2.3.6). Presumably, then, a grinder that would successfully grind Chromosorb would also grind sludge agglomerates, and the results of tests made on Chromosorb could be extrapolated to sludge agglomerates with a fair degree of confidence.

4.1 Materials

Two types of Chromosorb were used in the grinding tests — Chromosorb P and Chromosorb W. Both were acid-washed and were 30-60 mesh size. The particle density of Chromosorb W is 1.21 g/cm³, and the particle density of Chromosorb P is 1.35 g/cm³. The supplier of both Chromosorbs is Johns-Manville.

One of the "grinders" tested was a Gaulin 2F colloid mill, which was provided with piping for either recirculation or once-through flow. The gap opening between the rotor and stator was adjustable between 0.001 and 0.04 in. The throughput was about 100 gal/hr and was only slightly dependent on gap setting.

The other "grinder" tested was a Tekmar Super Dispax SD45 with a flow-through adapter. The adapter converted the unit to a flow-through configuration and, according to the manufacturer, permitted extrapolation of test results obtained with the small unit to results obtainable with the larger in-line grinders. The unit was equipped with a G-454 head and a regulator that permitted speed adjustment between 1,000 and 10,000 rpm.

4.2 Test Method

The particle density of Chromosorb is much less than that of Gunit tank sludge particles so that tests at the same weight fraction solids was not feasible. Instead, the tests were run with a volume fraction of Chromosorb in water approximately equivalent to the expected volume fraction of Gunit tank sludge. This volume fraction was 0.04 and was approximated by 50 g of Chromosorb in 1 liter of water.

Tests with the colloid mill were performed by setting the gap opening between the rotor and stator to the desired value, pouring the Chromosorb slurry into the feed tank, starting the unit, collecting the ground-slurry stream, and washing the colloid mill to remove residues. The particle size distribution of the ground slurry was then determined by a series of settling tests.

Tests with the Tekmar grinder were performed by adjusting the vertical elevation of a head tank so that the flow rate past the grinding head would be approximately 1 liter/min, which corresponds to a residence time near the head of 15 sec. The 1 liter of feed slurry was then poured into the head tank (which was agitated), and the grinder was run at the desired speed until the feed tank was empty. Residues were washed from the unit, and the particle size distribution of the ground slurry was determined by settling tests.

The equation for the unhindered settling velocity v of spherical particles in a fluid (Stokes' Law) is $v = [D^2 g_c (\rho_s - \rho) / 18\eta]$. If a slurry of

various size particles is shaken in a container and allowed to settle for a particle time, a rough separation by particle size will be achieved. The largest particles will settle out of suspension, the smallest particles will remain in suspension, and the intermediate-size particles will be partly in suspension and partly settled. This type of separation was used to determine the particle size distribution of the ground slurry. In a typical determination, the ground slurry was shaken in a 1-liter cylinder and allowed to settle for 15 sec. The unsettled slurry was poured off and held. The settled fraction was resuspended in fresh water, allowed to settle 15 sec, and separated into settled and unsettled fractions. The settled fraction was dried and weighed; the unsettled fraction was combined with the unsettled fraction from the first shaking, and the combined fractions were shaken and allowed to settle for 30 sec. This procedure was continued with progressively longer settling times until a fairly precise size distribution of the ground slurry was obtained.

4.3 Grinding Tests and Test Results

The tests run with the two grinders are summarized in Table 10.

Table 10. Summary of grinder tests

	Chromosorb	Setting	Throughput (liters/min)	Number of passes
Tekmar test 1	P	6,000	1.0	1
2	P	6,000	0.44	1
3	W	6,000	0.86	1
4	W	Max	0.88	1
5	W	Max	0.79	2
6	W	Max	0.74	4
7	P	Max	0.80	4
Colloid Mill Test 1	W	0.005	6.0	1
2	W	0.002	6.7	1
3	P	0.002	4.3	1

The effect of throughput rate is shown in Fig. 10; the effect of a twofold difference in rate is small.

The effect of grinder speed is shown in Fig. 11. The effect is appreciable in the size range of 100 to 200 μm but less marked for smaller or larger size particles.

The difference between Chromosorb P and Chromosorb W is shown in Fig. 12. The results obviously differ greatly with particles of different friability.

Figure 13 shows the effect of multiple passes through the Tekmar. Four passes through the test unit are reputedly equivalent to one pass through a three-stage unit.

Figure 14 compares the results of four passes through the Tekmar with one pass through the colloid mill (setting 0.005 in.). The results are approximately equivalent.

Figure 15 shows the effect of gap setting on colloid mill performance.

4.4 Conclusions

Either the Tekmar or the colloid mill proved capable of grinding a slurry of Chromosorb W from a 300- to 500- μm particle size to 70% less than 100 μm . Chromosorb W is less friable than any of the tank sludges and, unlike the sludges, does not contain a large fraction of 10- μm -size particles. It seems most probable, therefore, that either type of grinder would reduce sludge particle sizes to an acceptably small diameter.

5. TEST OF SUSPENDERS

The slurry resuspended from the Gunite tanks will be pumped through a 2-in. line that is several thousand feet long and has an irregular profile — one that has several "traps" for settled slurry particles. Figure 16 is a profile of this waste-transfer line. Preliminary tests with slurries of such materials as iron oxide, pottery clay, and fly ash indicated that the larger slurry particles would settle fairly rapidly from suspension, unless the slurry were constantly agitated. To avoid such settling, either the slurry particles would have to be ground to a very small size (10 μm or less) or suspenders (such as bentonite) would have to be used to

Fig. 10. Grinding test in Tekmar grinder - effect of throughput rate.

Fig. 11. Grinding test in Tekmar grinder - effect of grinder speed.

Fig. 12. Grinding test in Tekmar grinder - performance of different Chromosorbs.

Fig. 13. Grinding test in Tekmar grinder - effect of multiple passes.

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Fig. 14. Comparison of grinding performance of Tekmar grinder and colloid mill.

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Fig. 15. Grinding test in colloid mill - effect of gap setting.

Fig. 16. Profile of waste-transfer line.

keep the larger particles in suspension. A preliminary evaluation of commercially available suspenders was made to determine their applicability to the proposed system. In general, the tests made were settling tests (to determine the concentration of suspender necessary) and viscosity determinations.

5.1 Materials Tested

Suspenders are in wide use in the petroleum industry and are readily available from the suppliers to this industry. Baroid Petroleum Services kindly supplied test quantities of those drilling mud chemicals most likely to be useful in our application. Additional samples were obtained from Georgia Kaolin. Table 11 lists the chemicals and clays tested and gives some literature notes.

The material used to simulate sludge particles in the settling and viscosity tests included Fe_2O_3 , Fe_3O_4 , CaCO_3 , Al_2O_3 , pottery clay, fly ash, and various combinations of these materials. The approximate particle size of these materials was 40 μm or less, and the particle density varied from about 2.5 g to 5.24 g/cm³. Most of the tests were made with Fe_3O_4 , CaCO_3 , and combinations of the two. These two materials represented the range of particle densities likely to be found in the actual slurry, and their significant color contrast made differential settling comparatively easy to observe.

The effectiveness of bentonite and other suspenders is known to vary with both the ionic content and the pH of the fluid media. A preliminary analysis of the probable salt content of the actual resuspended slurry (based on 50 wt % solids with a particle density of 4.0 g/cm³ and an interstitial liquid composition the same as that of the ILW concentrate) indicated that the liquid phase would be 0.03 M NaNO_3 and 0.01 M NaOH . Most of the settling tests and the viscosity determinations were performed with solutions of this ionic content. Some tests were made with solutions of higher ionic content to investigate the effect of this variable.

5.2 Settling Tests

A series of preliminary settling tests was made with mixes of Fe_3O_4 , CaCO_3 , and bentonite in various proportions. We noted that selective settling of Fe_3O_4 would occur from mixes of 5 wt % Fe_3O_4 , 5 wt % CaCO_3 ,

Table 11. Clays and chemicals tested

Name	Material	Suggested concentration (%)	Notes
<u>Baroid Petroleum Services</u>			
Aquagel	Bentonite clay	3.0	Ineffective in high salt
Quickgel	Bentonite and polymer		
Zeogel	Attapulgate clay	5.0	Effective in high-salt solutions
Sea mud	Clay, thinner, and filtration agent		
Cellex	Sodium carboxymethyl cellulose	0.1	High or low pH
Q-Broxin	Lignosulfonate		A thinner; high or low pH
Barafos	Sodium tetraphosphate	0.1	A thinner useful to 5000 ppm chloride; optimum pH, 8-9.5
CC-16 (Carbonox)	Modified lignite		A thinner sensitive to Ca^{2+} or Mg^{2+}
B4-16 (Lo Loss)	Guar gum	0.5	Viscosity increaser
XC-polymer			
<u>Georgia Kaolin</u>			
Thixogel No. 2	Bentonite clay		Low viscosity; intermediate swelling
Thixogel No. 3	Bentonite clay		Low viscosity
Thixogel No. 4	Bentonite clay		Low viscosity; low swelling
Hydrite	Kaolinite		
Colloid BP	Montmorillonite		

and 2 or 3 wt % bentonite, but no selective settling occurred from mixes of 10 wt % Fe_3O_4 , 10 wt % CaCO_3 , and bentonite or from mixes of 5 wt % Fe_3O_4 , 5 wt % CaCO_3 and 5 wt % bentonite. Mixes with 2 wt % bentonite settled about 2% after 5 hr and about 15% after 24 hr; mixes with 3 wt % bentonite settled about 2% after 24 hr.

A second series of settling tests was made with a slurry of 5 wt % Fe_3O_4 and 5 wt % CaCO_3 to determine the effects of several suspenders at different concentrations and at high- and low-salt concentrations. The suspenders tested were Aquagel (bentonite from Baroid), Zeogel (attapulgitic from Baroid), and sea mud. The high-salt concentration was 0.09 M NaNO_3 and 0.03 M NaOH (three times the low-salt concentration). In both high-salt and low-salt solutions, the sea mud formed a lumpy suspension that settled 30% or more in 24 hr. The Zeogel was comparatively ineffective; in a 5 wt % concentration the slurry settled 28% in low-salt solution and 40% in a high-salt solution. The Aquagel was quite effective in low-salt solution (3 wt % settled less than 2% in 20 hr) and less effective in high-salt solution (3 wt % settled to about 30% of the original sludge volume after 22 hr). An increase in the concentration of Aquagel to 5 wt % increased the suspended volume to about 35% after 22 hr in the high-salt solution.

Another series of settling tests was made with a slurry of 5 wt % Fe_3O_4 , 5 wt % CaCO_3 , 3 wt % Aquagel, and various chemical additives. We observed that the addition of 0.5 wt % Q-Broxin, Barafos, or CC-16 to the slurry markedly increased the settling rate and that the Fe_3O_4 generally settled preferentially. The addition of 0.1 wt % Cellex to the slurry appeared to slow the settling rate in both high- and low-salt solutions, but there were some discrepancies in the data and some suggestion that the mixes containing Cellex were particularly shear sensitive. A test of 0.16 wt % X-C polymer in slurries with various concentrations of CaCO_3 (no bentonite) indicated that this additive affected slurry settling characteristics to about the same degree as 1 wt % bentonite.

A series of settling tests was made with 1 wt % bentonite and 5, 10, and 15 wt % of different solids for sludge simulants. Pottery clay, CaCO_3 , fly ash, and Al_2O_3 were used. The Al_2O_3 and the pottery clay did not settle appreciably in 20 hr at any concentration. The 15 wt % slurry of fly ash

settled about 6%; the lower concentrations did not settle. All concentrations of CaCO_3 settled very appreciably; the 5 wt % slurry settled to 30% of the original volume, the 10 wt % slurry settled to 50%, and the 15 wt % slurry settled to 64%. We concluded from this test series that the use of CaCO_3 and Fe_3O_4 (which settles even more easily) for sludge simulants would be conservative because the actual sludge particles will most likely be easier to keep suspended.

Tests were made with other possible suspenders — Hydrite, montmorillonite, and Colloid BP. None was as effective as bentonite. A test with sodium silicate indicated that it acted as a flocculant, not a suspender. A test with "super-slurper" (a colloidal starch) showed considerable suspending power, high sensitivity to chemical ions in solution, and a probable excessive viscosity.

On the basis of these tests (and viscosity determinations described in Sect. 5.3), we concluded that:

1. The sludge solids can be kept in suspension without agitation for at least several hours if a 2 to 3 wt % bentonite suspension is used and if the concentration of soluble salts is not excessive.

2. The concentration of soluble salts has a considerable effect. The suspending power of bentonite can be increased markedly by the addition of 0.01 M NaOH and decreased by the addition of NaNO_3 . An NaNO_3 concentration of 0.03 M has little effect; a concentration of 0.09 M has a more pronounced (but still tolerable) effect. Concentrations much greater than 0.1 M will probably require the use of a different suspender.

3. The use of additives does not appear promising. Cellex and X-C polymer are possible exceptions.

The results of settling tests with 15 wt % mixed solids (40% pottery clay, 10% CaCO_3 , and 50% Fe_3O_4) in a 2 wt % suspension of three different bentonites is shown in Fig. 17. A solution of 0.03 N NaNO_3 and 0.01 M NaOH was used in this test. The effect of the concentration of suspended solids on the settled volume of suspension after 24 hr is shown in Fig. 18; the effect of the concentration of soluble salts is shown in Fig. 19.

Fig. 17. Settling rates of 15 wt % solids with different suspenders.

Fig. 18. Settled volume after 24 hr with various concentrations of solids and suspenders.

Fig. 19. Effect of salt concentration on settled volume.

5.3 Viscosity Tests

Suspensions of interest to this project are non-Newtonian fluids (the shear stress is dependent on the rate of applied shear). In general, these suspensions have the characteristics of either a Bingham plastic or a power-law fluid. The characteristics of these two fluid types are quite different at low shear rates, but, at moderate and high rates of shear, both fluids behave as do power-law fluids (a plot of the shear stress and the shear rate is a straight line on log-log paper). The fluid-flow behavior of such fluids can be calculated if the slope and intercept of the shear stress versus shear rate plot are known; the determination of these values was the main objective of the viscosity tests.

A Fann viscometer was used in the tests. The particular model used had an R-1, B-1 rotor-bob combination and an F-1 spring; readings were taken at 3, 6, 100, 200, 300, and 600 rpm. The shear rate, in sec^{-1} , for this rotor-bob combination is 1.704 times the rpm; the shear stress, in lb/ft^2 , is the dial reading divided by 100. A line drawn through the experimentally determined values on log-log paper has a slope of n' (a measure of non-Newtonian behavior — water has an n' value of 1.0) and an intercept at unity rate of shear of K' (a measure of consistency; the higher the K' , the more viscous the fluid).

For convenience, the reading at 300 rpm is sometimes reported as the "viscosity" or "apparent viscosity" of the fluid. This value would be the viscosity if the fluid were Newtonian and had the effective viscosity at that particular rate of shear. It is a single number that is useful for comparative purposes. Hydrofracture grouts typically have an n' of 0.6, a K' of 0.01, and an apparent viscosity of 45 cP.

We determined the rheological properties of slurries of bentonite and CaCO_3 , Al_2O_3 , fly ash, and pottery clay. The results for 2 wt % bentonite and 15 wt % solids are shown in Fig. 20. These curves indicate that the fit of the points taken at low rates of shear is poor; this result suggests that these suspensions behave more like a Bingham plastic (with a minimum yield point) than a strict power-law fluid. The range of shear rates of most concern in the operation of the facility is between 200 and 400 sec^{-1} , however, and, in this region, the fit of the data points is good. The data

Fig. 20. Rheological properties of various solids.

also indicate that the data for CaCO_3 are quite odd. Further tests with CaCO_3 and bentonite suspensions showed that some of these suspensions are rheopectic — the shear stress increases as the suspension is sheared at a constant rate. This phenomenon was not observed when the bentonite concentration was 1 wt %. Substitution of CaSiO_3 , SrCO_3 , and MgCO_3 for the CaCO_3 showed similar, but less pronounced, effects with CaSiO_3 , some effects with SrCO_3 , and very pronounced effects with MgCO_3 . A suspension of 3 wt % bentonite alone showed some effects. A suspension of 10 wt % CaCO_3 , 5 wt % clay, and 3 wt % bentonite showed a slight rheopectic behavior; but, if either Fe_3O_4 or Al_2O_3 were substituted for the clay, the rheopectic behavior was very strong. Resolution of the factors affecting these phenomena seemed to be beyond the scope of this work and was not pursued further.

The effect of shear on viscosity was investigated by subjecting slurries of 10 wt % CaCO_3 and 2 wt % bentonite to varying degrees of shear and determining the rheological properties. The results are given in Table 12.

Table 12. Viscometer readings as a function of applied shear

	Reading (rpm)					
	3	6	100	200	300	600
10-sec blender	4	5	6	8	9	12
30-sec blender	5	5	7	9	10	14
1-min blender	6	7	9	11	13	18
5-min blender	11	11	25	30	33	36
2-min Tekmar	10	12	18	23	29	34
5-min Tekmar	9	12	46	54	57	64

Comparative tests were run with bentonite from different suppliers — Quickgel and Aquagel from Baroid and Thixogel from Georgia Kaolin. The solids used were a mix of 5% CaCO_3 , 45% pottery clay, and 50% Fe_2O_4 . The results obtained with 2 wt % Aquagel and 3 wt % Thixogel are shown in Fig. 21. The results with 2 wt % Quickgel were essentially identical to those obtained with Aquagel. On the basis of these tests, we concluded that Thixogel is the best suspender for the sludge resuspension process. The suspending power is slightly less, but the apparent viscosity is appreciably less.

Fig. 21. Rheological properties of bentonites.

The rheological properties of concentrated suspensions of Thixogel and Quickgel were determined (Table 13).

Table 13. Viscometer readings for concentrated bentonites

	Conc. (wt %)	Reading (rpm)					
		3	6	100	200	300	500
Thixogel,	4.8	2	2	3	4	6	9
	9.1	2	2	4	7	9	16
	13.0	3	4	11	18	24	40
	16.7	12	15	30	44	56	89
	20.0	29	31	53	75	130	174
Quickgel,	1	2	2	3	4	5	7
	2	3	3	6	8	9	15
	3	8	9	20	25	30	40
	4	14	24	43	54	60	75

6. GROUT MIXES

The sludge now in the Gunitite waste tanks will be resuspended and pumped to the new ILW storage tanks in Melton Valley. This resuspended sludge will subsequently be pumped to the new shale-fracturing facility and mixed with a cement-base mix to form a grout, which will be injected underground. This grout must have properties not greatly dissimilar from those of current shale-fracturing grouts. It must be fluid (an apparent viscosity of about 40 cP), remain fluid for at least 24 hr, exhibit phase separation of less than 5%, and have an acceptable compressive strength and leach rates for radionuclides. Each of these properties could be drastically affected by relatively small changes in feed composition, and, because the substitution of a thick slurry for an NaNO_3 solution is a major change, preliminary tests of the compatibility of the waste slurries and cement-base mixes were needed. We made these preliminary tests with slurries of Fe_3O_4 , CaCO_3 , and bentonite and measured the viscosity and phase separation of the grout mixes; determinations of compressive strength and leach rates, however, were not made at that time.

6.1 Test Procedure

The test methods developed for the shale-fracturing process were used in this series of tests. The components of the dry solids mix were blended by shaking in a covered jar. The slurry was put in a Waring blender, and the desired weight of blended solids was added over a 15-sec interval with the blender running at 2000 rpm. The grout was then stirred for an additional 15 sec at 5000 rpm. This procedure was designed to shear the grout sample approximately the same as the grout is sheared in the shale-fracturing facility.

Phase separation of the grout was measured by pouring a grout sample into a 250-ml graduated cylinder and measuring the volume of free water above the grout layer after 3 hr. The apparent viscosity of the grout was determined with a Fann viscometer (see Sect. 5.2).

6.2 Tests

Tests were made with the standard hydrofracture dry mix and with five variants of this mix. The composition of all mixes tested is given in Table 14.

Table 14. Compositions of dry solids mixes (wt %)

	Standard	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Cement	38.5	42	44	46	46	46
Fly ash	38.5	42	44	46	46	46
Attapulgate	15.4	8	4.5	0	0	0
Pottery clay	7.7	8	7.5	8	8	8
Retarder	0.05	0.05	0.05	0.05	0.10	0.20

The tests made and the results of these tests are summarized in Table 15.

The results of the first four tests indicated that an acceptable grout could be formed from a sludge slurry of 10 or 20 wt % solids and the standard dry mix if the mix proportion were no more than 4 lb/gal.

Table 15. Tests of cement mixes

Test	Mix	Sludge solids concentration ^a (wt %)	Suspender (%)	Mix ratio (lb/gal)		Grout properties	
				Dry mix	Total solids	Phase separation (%)	Apparent viscosity (cP)
1	Std	20	None	4	5.9	1.6	45
2	Std	20		3	4.9	2.4	21
3	Std	10		4	5.0	3.2	17
4	Std	10		3	4.0	12.0	21
5	1	20		5	6.9	12.0	12
6	1	20		6	7.9	4.0	17
7	1	20		7	8.9	1.6	36
8	2	20		7	8.9	3.6	20
9	2	20		8	9.9	0.8	49
10	2	20		9	10.9	0.6	77
11	1	10	3	2.81	4.0	3.0	19
12	1	10	3	3.81	5.0	2.0	38
13	1	10	3	4.81	6.0	1.0	54
14	3	10	3	5.81	7.0	6.0	13
15	3	10	3	6.81	8.0	3.6	15
16	3	10	3	7.81	9.0	1.6	19
17	3	10	3	8.81	10.0	0.4	27
18	3	10	3	9.81	11.0	0	36
19	4	10	3	8.81	10.0	2.0	20
20	4	10	3	9.81	11.0	0	31
21	5	10	3	8.81	10.0	3.0	13
22	5	10	3	9.81	11.0	0	20

^aSolids were equal weights of Fe₃O₄ and CaCO₃.

The results of tests 5 through 10 indicated that the proportion of attapulgite in the dry mix could be decreased substantially if the mix ratio were increased to about 7 or 8 lb/gal. These results suggested a correlation between the amount of suspender in the grout and the quantity of other solids in the grout. Tests 11 through 18 were made to check the validity of this correlation. Figure 22 shows the suggested correlation — weight percent suspender (bentonite or attapulgite) in the grout plotted against pounds of total solids (exclusive of any suspender either in the dry mix or in the sludge) used per gallon of sludge. The three points used to establish the correlation were obtained from shale-fracture data and tests 5 through 10.

Figure 23 shows the suggested correlation of Fig. 22 with the sum of the mix ratios of tests 11 through 18. The correlation is good for the conditions of tests 14 through 17; it suggests that test 17 should have acceptable properties, which is the case. The correlation is not good for tests 11 through 13; it suggests that more solids are needed than was the case. Allowance should probably be made for the difference in effectiveness of bentonite and attapulgite.

Tests 19 through 22 were made to investigate the effect on grout properties of a larger amount of retarder. We found that increasing the concentration of the retarder reduced the grout viscosity but increased the phase separation. The mix ratio would have to be increased to obtain an acceptable phase separation, and this increase would also result in a higher viscosity. The net effect would be a grout with essentially similar properties but with higher dry solids consumption. We concluded that there is little incentive to pursue this approach further. Setting time with mix 5 was between four and seven days.

6.3 Conclusions

Slurries of simulated sludges are apparently compatible with a number of relatively simple modifications of the standard shale-fracturing mix. If a suspender is used in the slurry (as appears likely), much less suspender is required in the dry solids blend. The total amount of suspender required is a function of the amounts of cement and fly ash used, and some trade-offs are probably feasible. A correlation for estimating this trade-off is suggested.

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Fig. 22. Correlation of mix ratio with suspender concentration.

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Fig. 23. Fit of correlation with experimental points.

7. TESTS ON ACTUAL SLUDGE

Sludge samples were taken from tanks W-5, W-6, and W-8, and a series of tests was run to determine the physical properties of the sludge particles. The effectiveness of the Tekmar grinder and bentonite suspenders was also evaluated. These tests confirmed that the behavior of the actual sludge particles was similar to that of the synthetic sludge particles used in the tests discussed in Sects. 3.0-6.0.

7.1 Samples

Samples of sludge were taken from tanks W-5, W-6, and W-8. A river-bottom sampler (capacity ~400 ml) was used; in each case the sample was taken by pushing the sampler through the sludge layer to the bottom of the waste tank. The sample obtained would therefore not be typical of the sludge at any particular depth in the tank.

The sample from W-5 was gray and "sandy." The sample size was comparatively small (<100 g dry weight). The sample from W-6 was red and "sticky." Not much sample was obtained (~100 g dry weight). The sample from W-8 was gray-brown and "stiff." A full sample was obtained.

7.2 Density Measurements

The weight fraction of solids in each sludge sample was determined by weighing a sample, washing it several times to remove soluble components, drying the residue, and weighing. A similar procedure is described in Sect. 2.3.2. Three samples were taken from the bulk sample — at the top, middle, and bottom. These values are given in Table 16 for tanks W-6 and W-8; the values obtained for tank W-5 were distorted by a malfunctioning balance and are not regarded as meaningful. The results obtained for tank W-8 indicate that a large fraction of the sludge in this tank is composed of soluble components; the measured weight of solids decreased by about 30% after washing. The samples from W-6 also show a decrease after washing, but only of about 10%.

Density of the sludge solids was determined from a composite sample of each sampler load. The composite samples were washed, dried, and weighed.

An equal weight of water was added, and the volume of the resulting mixture was determined. A relatively large sample (~17 g of dry solids) was used to minimize errors of measurement. These density values are also given in Table 16.

Table 16. Density and solids percentage of sludge samples

	Solids density (g/ml)	Solids (wt %)	Solids after wash (wt %)
W-5	3.8		
W-6 Top		29.2	25.5
Middle	1.92	35.1	31.4
Bottom		42.8	38.0
W-8 Top		37.2	24.4
Middle	2.30	33.9	20.5
Bottom		37.3	22.1

7.3 Particle Size Determination

An approximate particle size distribution was determined for a sludge sample from each tank. This distribution was determined so that the grinder performance could subsequently be evaluated. In this determination a small sample (5 to 6 g) of dried sludge was suspended in 500 ml of water, and the fractions of solids settling in 30 sec, 1 min, 15 min (or 20 min), and overnight were determined. These fractions are proportional to the size and density of the solids particles and can be used to calculate an approximate size distribution. This test method is described in Sect. 4.2. The results of these tests are given in Table 17. The results indicate that 80 to 90% of the particles in all three samples are larger than 60 μm . The results are at variance with earlier tests on other samples, which indicated a high fraction (>50%) of particles smaller than 10 μm in each of the three waste tanks (Sect. 2.3.3). This difference could result from atypical samples, either in this determination or previously. An alternative explanation is that the drying procedure (used on the samples taken in 1977 but not on earlier samples) caused agglomeration of the smaller

particles and biased the results. The soluble fraction of the sludge sample observed in these tests (~5 to 10%) was smaller than the soluble fraction observed in the earlier tests on the same sample. No reason for this difference (apart from sample variability) is known.

Table 17. Particle size distribution

	W-5	W-6	W-8
Fraction settling in 30 sec	0.798	0.858	0.824
Calculated particle diameter, μm	62	98	82
Fraction settling in 1 min	0.065	0.043	0.040
Calculated particle diameter, μm	44	69	58
Fraction settling in 15 min (20 min)	0.042	(0.042)	(0.028)
Calculated particle diameter, μm	11.4	15.5	13
Fraction settling overnight	0.008	0.015	0.007
Calculated particle diameter, μm	1.4	2.2	1.9
Soluble fraction	0.086	0.042	0.102

7.4 Grinding Tests

Grinding tests were made on each of the sludge samples to determine the effectiveness of the Dispax grinder-emulsifier in this application. The samples processed by the Dispax unit were subsequently used for tests of suspending agents (Sect. 7.5).

The Dispax unit used in these tests was a model SD45K with a G-456 generator head. It was similar to the unit used for grinding tests with simulated sludge (Sect. 4) except that the generator head was designed for handling smaller particles. The unit was provided with a flow-through system attachment that limited product holdup time near the generator head and simulated the action of larger in-line units that will be installed at the resuspension facility. Tekmar Company (the supplier of the unit) recommended three passes through the system, with a dwell time of about 15 sec per pass to simulate the expected performance of an in-line unit. Previous grinding tests with this equipment using Chromosorb W to simulate sludge solids (Sect. 4) had shown a progressive decrease in particle size with each pass through the machine. After three passes through the unit,

the percentage of Chromosorb particles smaller than 100 μm had increased from an original value of 15% to 65%. Earlier tests had indicated that Chromosorb particles were more resistant to size reduction than were sludge particles, and a more complete size reduction was expected in these tests.

The procedure followed in these tests was essentially identical for samples from W-5 and W-6. It was modified somewhat for the samples from W-8 because of problems associated with the relatively high fraction of solubles in the W-8 sample; this modification is discussed later. For the tests on both the W-5 and W-6 samples, about 20 g of dry solids were used. These solids were suspended in 1 liter of water and passed through the grinder. The ground solids were dried, and a particle size determination was made. These solids were resuspended in water, passed through the grinder a second time, and dried. A second particle size determination was made. The solids were then ground a third time, and a third particle size determination was made. These solids were then used for suspension tests (discussed below). The results of these tests are given in Table 18.

The same procedure used with the samples from W-5 and W-6 was attempted with the sample from W-8. Approximately 30% of the sample was not recovered, however. This sample loss (assumed to be soluble components) complicated the interpretation of the results; consequently, the procedure was modified slightly. A fresh sample from W-8 was washed three times to remove solubles and was subsequently dried. This washed sample was passed three times through the grinder. The ground sample was then dried, and a particle size determination was made. No particle size determinations between grindings were made with this sample. The results are given in Table 18.

The results do not show that the grinder had any appreciable effect on the particle size distribution. The limitations of the particle size distribution determination are such, however, that no distinction is made between particles larger than about 70 μm . Quite possibly, therefore, the largest particles were attenuated in the grinder, but the size determination procedure was not sensitive enough to detect this change. The suspension tests (Sect. 7.5) on ground and unground samples from W-8 support this belief. In these tests, a significant fraction of the solids settled out of the suspension of unground particles, but nothing settled

Table 18. Particle size distribution after grinding

	W-5	W-6	W-8	W-8A
<u>First grinding</u>				
Fraction settling in 30 sec	0.848	0.575	0.558	
Calculated particle diameter, μm	62	98	82	
Fraction settling in 5 min	0.106	0.088	0.020	
Calculated particle diameter, μm	19.7	31	26	
Fraction settling in 20 min	0.034	0.059	0.010	
Calculated particle diameter, μm	9.8	15.5	13	
Fraction settling in 16 hr	0.012	0.117	0.105	
Calculated particle diameter, μm	1.4	2.2	1.9	
Soluble		0.161	0.307	
<u>Second grinding</u>				
Fraction settling in 30 sec	0.786	0.596		
5 min	0.167	0.132		
20 min	0.043	0.082		
16 hr	0.005	0.158		
<u>Third grinding</u>				
Fraction settling in 30 sec	0.854	0.663		0.754
5 min	0.112	0.091		0.036
20 min	0.031	0.064		0.036
16 hr	0.003	0.165		0.075
Soluble				0.099

out of the suspension of ground particles. We concluded, therefore, that the grinder is likely to be effective in grinding particles to an approximate diameter of 100 μm but that it is ineffective in grinding smaller particles.

7.5 Suspension of Sludge

The samples of sludge from W-5 and W-8 used in the grinding tests were also used in the suspension tests. In addition, one sample from

W-8 was used directly — without grinding pretreatment. In all cases, about 10 g of dried solids were suspended in about 500 ml of a 1 wt % suspension of Quickgel (bentonite plus polymer) in water. The level of settled solids was then noted at various time intervals. Sufficient Quickgel was added to bring the suspension concentration to 2 wt %, and the settling test was repeated. More Quickgel was added to bring the suspension concentration to 3 wt %, and the settling test was repeated.

The results of the tests for W-5 and W-8 are shown in Fig. 24. These results are essentially the same as those obtained with a synthetic sludge (for the same suspender, solids concentration, and solution ionic concentration). This similarity of results obtained with synthetic and actual sludge strongly suggests that the synthetic sludges are suitable substitutes for the actual sludges and that the results of tests made with the synthetic sludges can be extrapolated to the actual sludges with reasonable confidence.

We observed that, in the tests performed on the unground sample of W-8 in 2 wt % Quickgel, an appreciable fraction of the particles settled out of suspension. This phenomenon was not observed in samples that had been ground; in these cases the suspension appeared uniform from top to bottom. Most probably, the grinding operation breaks up the largest particles — those that would have the strongest tendency to settle from suspension.

7.6 Analysis of Soluble Fraction

Some of the work done on the sample from tank W-8 had indicated that a large fraction of the sludge in this tank could be composed of material that was soluble in water. The presence of such material could affect the process in several ways — by decreasing the effectiveness of the suspender, by increasing the slurry viscosity, or by affecting the compatibility of the slurry with the cement mixes. Accordingly, the amount and analysis of the soluble material were determined for one sample of sludge.

The sample of sludge from W-8 was dried, weighed, and slurried in water. The supernatant was analyzed; the solids were dried, weighed, and reslurried in water. Again, the supernatant was analyzed. The loss of material in the washings was 5.8 wt % of the original sample. The loss was partly soluble material and partly very fine solid particles that had

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Fig. 24. Settling tests on sludge samples.

settled very slowly. The major chemical species were carbonate, sulfate, and nitrate ions; very small quantities of chloride and phosphate ions were observed. The concentration of nitrate ions in the first wash solution was 0.026 M — very close to the 0.03 M postulated for the evaluation tests for suspenders.

8. FIELD TESTS

A series of field tests has been made to simulate the operations of sludge resuspension and transfer and to evaluate the problems encountered. A mockup of a part of one of the Gunitite tanks has been built, and a slurry of mixed shale and clay has been used to simulate the sludge. Various pump and sluicer configurations have been tested. Tests of several different types of instruments have also been made to evaluate these instruments for possible use with slurries.

8.1 Mockup Description

The sludge pumping test facility consists of (1) a semicircular concrete tank the same size and configuration as one-half a Gunitite waste tank bottom, (2) a pump platform the same distance above the bottom of the concrete tank as the ground level is above the bottom of the Gunitite tanks, and (3) a set of pumps and sluicing equipment. The concrete tank is 4 ft deep and has a radius of 25 ft. A partition in the middle of the tank divides the tank into equal-size north and south compartments. The pump platform is at the corner of the south tank compartment; the floor of this platform is 20 ft above the bottom of the concrete tank. The available pumps include (1) a two-stage No. 8 Moyno pump; (2) a Super Dispax dispersing pump (grinder) manufactured by the Tekmar Company, model T 150/4L; (3) a submersible, vertical centrifugal pump manufactured by the Midland Pump Company, model AFP-004; and (4) a positive displacement pump manufactured by the Waukesha Company, model 125 D1. Flexible lines were used to make connections between the various pumps, which were altered for different tests; not all pumps were used for all tests.

The Moyno pump and Super Dispax grinder were mounted on the pump platform 20 ft above the concrete tank bottom. The vertical centrifugal pump

was suspended from a chain hoist located in the south corner of the concrete tank next to the pump platform. The Waukesha positive displacement pump was mounted on a separate steel platform about 3 ft above the concrete tank bottom. A simulated sludge was prepared from a mixture of 25% pottery clay and 75% shale. This mixture was added to the south tank compartment to a depth of 1 ft and sluiced to the inlet of a diaphragm pump (2-in. Sandpiper, model 2A2-A, manufactured by Warren Rupp Company). The diaphragm pump picked up the water-solid mixture and forced it through the Super Dispax grinder to the Moyno pump. The Moyno pump discharged into the north tank compartment. This operation required approximately 77 hr. The resulting shale-clay mixture consisted of two fractions. One fraction was made up of particles about 1/4 in. in diameter and represented approximately 25% of the total solids. The second fraction (75% of the total solids) consisted of a range of particles, of which some settled immediately and some required several days to settle. The average depth of clay in the tank compartment is about 6 in. after standing for several weeks or 8 to 10 in. after settling for 48 hr.

During the grinding operation, we experienced considerable difficulty with line and pump plugging. Three screens of different design were fitted to the intake line to prevent the entrance of large solids. The first and second designs consisted of lengths of 2-in. pipe having 1-in. holes drilled in rows. The first design was 6 in. long; the second was 12 in. long. These designs failed. The final design consisted of a box fashioned from floor grating with dimensions of about 12 x 12 x 18 in. This design also proved to be unsatisfactory. Large chunks of shale accumulated around the intake screens, shut off the flow, and necessitated manual removal of the shale chunks with a shovel. We decided that it was easier to abandon the screens and disassemble the lines and pumps when plugging occurred.

8.2 Tests at Mockup

The first four tests were run to determine the effectiveness of a water jet in dislodging and suspending clay in the concrete test tank. Water was pumped from the north tank compartment by the Moyno pump to a hand-held hose nozzle located on the top of the pump platform (Fig. 25).

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Fig. 25. Equipment arrangement for initial sluicing tests.

This operation produced a flow rate of 55 gal/min through a 0.5-in. nozzle at a pressure of 120 lb/in.². The water jet was directed back and forth over the entire surface area of the south tank compartment, keeping the clay in suspension and continuously stirring up fresh clay. The vertical centrifugal pump was used to pump the clay-and-water slurry to the north tank compartment. The pumping rate of the centrifugal pump was controlled by a throttling valve at the end of the discharge line, which balanced the centrifugal pump and Moyno pump discharge rates and held the water level constant in both tanks.

In tests 1 and 2, the water level in the south compartment was held at two different heights to determine the relative effectiveness of the water jet when penetrating water layers of two different thicknesses. Samples of the Moyno pump intake and centrifugal pump discharge were taken at 15-min intervals and dried in the laboratory to determine the solids content. The results are given in Table 19.

In these tests, the concentration of solids in the slurry pumped from the south compartment was increased twofold by dropping the water level from 10 in. to 6 in. This operation demonstrates the necessity for maintaining the liquid inventory in the tank at a minimum if the effectiveness of the jet stream is to be fully realized.

Settling rate determinations were made on two samples of suspended solids during test 1. The results (Table 20) mainly illustrate the problem of getting typical samples.

In test 3, 100 lb of bentonite and 500 lb of attapulgite were added to the south tank compartment to aid in holding the clay in suspension. The pumping-and-sluicing operation was carried out as in tests 1 and 2. Samples were taken at 15-min intervals at the intake of the Moyno pump and discharge of the centrifugal pump. The results are given in Table 21.

Test 4 was a repetition of test 3, except that the contents of the north tank compartment were thoroughly mixed before starting the test. The results of the test are given in Table 21.

The results of these tests indicated that even a relatively small amount of suspender (1% attapulgite and 0.2% bentonite) will greatly increase the concentration of sludge particles in the process stream. However, the suspender must be well mixed (as it evidently was not in test 3).

Table 19. Solids concentration entering and leaving tank — tests 1 and 2 (g/100 ml)

Sample No.	Inlet concentration	Exit concentration
<u>Test 1 — water level in south compartment, 10 in.</u>		
1	0.06	2.11
2	0.16	3.61
3	0.12	3.79
4	0.14	2.87
5	0.43	2.92
<u>Test 2 — water level in south compartment, 6 in.</u>		
1	0.06	4.23
2	0.45	8.00
3	0.34	6.10
4	0.93	7.31
5	1.17	8.39

Table 20. Particle size distribution of pumped sludge

Particle size (μm)	Sample 1 (%)	Sample 2 (%)
500 and larger	7.5	33.6
150 to 500	4.6	10.0
40 to 150	7.1	20.1
15 to 40	61.6	32.7
4 to 15	19.2	3.6

Table 21. Solids concentration entering and leaving tank — tests 3 and 4 (g/100 ml)

Sample No.	Inlet concentration	Exit concentration
<u>Test 3</u>		
1	1.5	5.7
2	3.1	5.4
3	3.7	9.6
4	4.6	11.0
5	4.0	6.9
<u>Test 4</u>		
1	12.41	13.39
2	12.16	13.64
3	9.66	14.43
4	7.38	15.15
5	12.88	15.85

Test 4 demonstrated that a slurry containing a suspender can be recirculated until the desired solids concentration is reached.

In test 5, the contents of the north tank compartment were agitated for 1.25 hr. The concentration of the solids in the nozzle discharge increased until it reached 19.5 wt %. This operation demonstrated that bentonite can be used to suspend the solids and hold them in suspension until the acceptable concentration is reached.

Tests 6 and 7 were run to evaluate the position of the sluicing nozzle in the tank. In these tests, water was pumped from the north tank compartment by using a positive displacement pump (Waukesha 125D1, 55 gal/min, 120-lb pressure) to a 0.5-in. sluicing nozzle directed on the clay-shale layer in the south tank compartment (Fig. 26). In test 6, the nozzle was mounted on a 6-ft length of 2-in. pipe and hand-held about 2 ft off the tank bottom, whereas, in test 7, the nozzle was an integral part of a mud gun mounted on the tank center about 7 ft above the tank bottom. The clay-shale suspension created by the jet action was pumped through the vertical centrifugal pump, the Super Dispax, and the Moyno pump to the north tank

Fig. 26. Equipment arrangement for later sluicing tests.

compartment. In test 6, with the nozzle located on the tank sidewall about 90° around the tank from the pump suction, complete removal of the clay-shale mixture was accomplished in 7 hr.

In test 7, using the centrally mounted mud gun, 10 hr was consumed, with a considerable quantity of the material left in the tank. As the test progressed, heavy solid particles collected in a band next to the tank sidewall. Particles near the pump intake were easily transported by the jet and pumped out, but, as the distance along the sidewall increased, the heavier particles became progressively more difficult to move to the pump intake. Particles near 90° from the pump intake could be moved only with a great deal of wasted motion.

8.3 Instrument Tests

Several process control instruments were purchased and installed in the pump test facility for evaluation. These instruments included (1) a vibrating reed densimeter, manufactured by the ITT Barton Company; (2) a falling-piston-type viscosimeter, manufactured by the Norcross Company; (3) a sludge density controller, manufactured by the National Sonics Corporation; (4) a target-type flowmeter, manufactured by the Foxboro Company; (5) a portable sonic flowmeter, manufactured by Scarpa Laboratories, model SFM-3B-BP; (6) a counterflow meter (Sect. 3.2); and (7) a vibrating rod viscometer, manufactured by Automation Products, Inc.

8.3.1 Target flowmeter

The sensing element of the target flowmeter consists of a round plate supported in the center of a short pipe section by a force bar connected to a differential pressure transmitter. The force exerted on the round plate by the flowing stream is sensed by the differential pressure transmitter, which transmits a readout signal proportional to the square of the liquid flow rate.

This instrument appeared to operate satisfactorily in the range of 40 to 70 gal/min, with the output signal directly proportional to flow rate. Flow rate measurements were made with line pressures up to 60 lb/in.². The instrument was operated on water and a clay-iron oxide slurry with a density of 1.140. The only difficulty experienced with this instrument

was caused by small rocks or shale particles lodging in the open space between the target and pipe sidewall, which happened on one occasion. On another occasion, a small pebble lodged between the force rod and the housing. Because a part of the opening (between the target and pipe) was closed by the obstruction, a high reading resulted, and the instrument did not return to zero when the flow was shut off. This difficulty should not constitute a serious problem in the Guniting tank sludge removal operation because the entire flow will pass through a grinder, which should reduce the size of solids particles below the size that would hang the flowmeter. If high-accuracy flow-rate measurements are desired, provision should be made to bypass the meter and to back-flush it with clean water. The performance curves (Fig. 27) show that, for a given flow rate, the transmitter output for clay is about 10% higher for a density of 1.140 than for pure water.

8.3.2 Sludge density controller

A model 494T sludge density controller, manufactured by National Services Corporation, was purchased and installed in the 2-in. discharge line of the Moyno pump at the sludge-pumping test site. This device is used a great deal in sewage-disposal systems for sensing sludge density and for generating an appropriate control signal to a pump. It consists of an ultrasonic transmitter and receiver mounted in a 4-in. pipe section and an electronic control circuit.

A sound wave is beamed by the transmitter through the liquid layer to the receiver on the opposite side. A change in the solids concentration in the intervening liquid causes a change in the attenuation of the sound beam. This change is picked up by the electronic control circuit, which activates a relay that is used to start and stop the pumps. A change in the gain setting (a variable resistor with a graduated dial) in the electronic circuit permits the selection of the sludge consistency at which the pump relay operates. Clay slurries with different solids concentration were introduced into the instrument, but the range of the instrument was not great enough to handle solids having concentrations of more than about 10%. Because sludges containing 15 to 20% solids are expected to be encountered in the Guniting tank operation, it was necessary to increase the instrument range. The sonic and electronic circuitry were removed from

Fig. 27. Performance curve for target flowmeter.

the 4-in.-pipe section, and a new 2-in. stainless steel sensing cell was constructed according to advice from manufacturer representatives. Subsequent tests of the new instrument indicated that a decrease in instrument range occurred, rather than the expected increase. This decrease indicated that a larger, rather than a smaller, section should have been selected, perhaps an 8-in.-diam section. This use of a larger section would represent problems in the piping system, however, because the resulting drop in liquid velocity might cause solids to settle out of suspension. Further work is not currently planned for this instrument.

8.3.3 Vibrating rod viscosimeter

A vibrating rod viscosimeter (model CL-10DV-2; trade name, Dynatrol), manufactured by Automation Products, Inc., was operated unsuccessfully in the hydrofracture system to measure the viscosity of the cement grout. The Brown recorder and electronic signal converter of this instrument were moved to the sludge pump test site. A new vibrating rod sensor was purchased and installed in the slurry tank. A sample cell of pipe fittings was fashioned around the horizontally mounted sensor. Several clay slurry samples of known composition were placed in the sample cell for measurement. The output readings increased linearly as the solids content of the samples increased but gave inconsistent results when the solids content was decreased. The instrument was sent to the Instrument and Controls Division shop for checking but was found to be in perfect working order.

The sensor was reinstalled in the slurry tank, and tests were made to analyze the problem. We found that the reading of the viscosimeter gradually increased when immersed in an unagitated tank, indicating that the small solids deposit clinging to the sensing rod element was producing a high readout value. This finding was substantiated when air was bubbled around the sensor to dislodge the solids collected on it.

One of the two possible uses of the viscosimeter is to detect the interface if solids prematurely settle out in Guniting tank W-10, which will be used for collecting and storing the sludge during the Guniting tank sludge-removal operations. The applicability of the instrument for this purpose was verified by lowering the sensor (mounted horizontally) through a 10-in.-thick water layer suspended on top of a clay-iron oxide slurry. The sensor

was lowered gradually through the water layer, with readings being taken at 2-in. intervals. The reading on the Brown recorder was 1.3 mV when the sensor was in the water layer, was 4.0 mV when the sensor was in the slurry layer, and returned to 1.3 mV when the sensor was retracted into the water layer and the clinging sludge was removed from the sensor by swishing it through the water.

The viscosimeter was installed in the discharge line of the Moyno pump where this line entered the slurry mixing tank. The sensing element was screwed into a 1.5-in.-pipe tee pointing vertically downward so that the upward flow of slurry would wash the sensing element. Preliminary measurements indicated that the instrument was sensitive to flow and that it would be necessary to reduce the flow past the sensor or to take a sample periodically and measure the viscosity under static conditions. Further work will be necessary to establish the operating requirements and to calibrate this instrument against known slurry concentrations.

8.3.4 Counterflow meter

The counterflow meter was tested in a pump test loop (Sect. 3.2) and found to give a reliable indication of slurry density and either viscosity or flow rate. In the pump test loop the necessary pressure-drop measurements were made with manometers that were directly connected to the slurry lines. Such a readout would not be practicable for field use with radioactive slurries; an alternative system would be required. All of the field development work with this device was therefore directed toward the evaluation of readout devices for small pressure differences.

A vertical 2-in.-pipe section in the shape of a "U" was installed in the discharge line of the vertical centrifugal pump. Two pressure sensors (Fig. 28) were installed in each side of the U, with a distance of 60 in. between centers (Fig. 29). A differential pressure transmitter was installed for each side of the U. On the left side of the U the lower sensor was connected to the high-pressure side of the pressure-sensing diaphragm in the transmitter, and the upper sensor was connected to the low-pressure side of the diaphragm. The same connections were made for the differential pressure transmitter on the right side of the U. The transmitters and

Fig. 28. Arrangement of pressure sensor.

Fig. 29. Test arrangement for counterflow meter.

lines connected to the sensors were filled with kerosene. Water was pumped through the U section downward on the left side and upward on the right side.

The output of the transmitter on the left side of the U should have indicated a pressure drop equal to the difference in the friction head and the static head. The output of the transmitter on the right side of the U should have indicated a pressure drop equal to the sum of the friction head and the static head. With this information, it should have been possible to calculate both the friction head and the static head. However, the values of the flow rate that were calculated from the friction head measurements disagreed considerably with the actual measured values. This difficulty was attributed to insensitivity in the pressure sensors. When the tests were repeated with the pressure-sensor inserts removed and the differential pressure transmitters replaced with manometers, reliable and consistent values were obtained.

An electronic transducer, manufactured by the Setra Systems, Inc., of Natick, Mass., was tested as a possible alternative pressure-drop measuring device. Setra model 237 was chosen because of its flush mounting and small diameter (0.5 in.). Two model 237 transducers were procured along with a model 300A digital readout. The two transducers were installed in a 1-in. pipeline, with a 5-ft spacing. Initial operation indicated unacceptable sensitivity to the flow pulsations created by the Moyno pump. When operated on the plant water supply, the output was quite improved, but still unreliable. With further operation, the instrument performance became quite erratic. A check of the instrument by the Instrumentation and Controls Division revealed that one of the transducers had failed. Current plans do not call for further work on this instrument.

8.3.5 Other instruments

The densimeter, viscosimeter, sludge density controller, and target flowmeter were installed in the discharge line of the Moyno pump. A 300-gal tank equipped with an agitator was installed in the system to provide a small volume of slurry of known concentration.

The vibrating reed densimeter utilizes the principle that the frequency of vibration of a reed immersed in a liquid is dependent on the density of the liquid. The electronic circuit senses the frequency and converts this

signal into an electronic display of density. This instrument was found to be quite sensitive to ambient temperature changes and to vibration from the Moyno pump.

The falling-piston viscosimeter was found to be unsatisfactory for our application because of (1) lack of clearance between the piston and the cylinder well in which it operates, (2) an unreliable sensing magnet mounting, and (3) the sensitivity of the instrument to density changes in the liquid being monitored.

The portable sonic flowmeter consists of an electronic circuit housed in a portable metal box with a flexible lead attached to a sonic detector, which can be clamped to a pipeline. The sound created by the rapid movement of liquid in the pipeline is detected electronically and read on a meter mounted on the face of the box. The sonic flowmeter sensing element was clamped on the Moyno pump discharge line about 25 ft from the pump. The signal generated by a 65-gal/min flow rate through the 2-in. pipe was not large enough to be detected by this model.

8.3.6 Valve testing

Three valves (two pinch valves and a ball valve) were installed in the Moyno pump discharge line. A clay-iron oxide slurry with a density of 1.140 g/cm^3 was pumped through the valves at a flow rate of about 50 gpm. The two 2-in. pinch valves were operated by air pressure. The pinch valves were manufactured by ITT Grinnel Corporation and by RKL Controls, Inc. The ITT valve was equipped with spring closure; the RKL valve used residual air pressure for closure. Both valves were equipped with a Moore positioner for throttling capability. A 3-in. Worcester ball valve was operated manually. A test pressure of 30 lb/in.^2 was used periodically to check for leakage. The ITT Grinnel valve operated in excess of 922 cycles without leakage, and the RKL valve, in excess of 860 cycles. The RKL valve never functioned quite satisfactorily, and disassembly revealed a ruptured operator diaphragm. Failure of the diaphragm is not surprising because it contained only one layer of fabric reinforcing mesh. Apparently the diaphragms are made unusually thin to provide the high degree of flexibility required in the operation of the rolling diaphragm (Rollomotor).

9. CONCLUSIONS

On the basis of the tests described in this report, we have reached the following conclusions:

1. Resuspension of the sludge with a water jet is quite feasible. This operation is much easier when using a bentonite-water suspension for reslurrying than when using water.

2. Sludge particles can be ground to a size small enough to suspend readily in bentonite.

3. Bentonite suspensions (2 to 3 wt %) will be adequate, unless the salt content is excessive. The "viscosity" of these suspensions will not be excessive.

4. The suspensions can be made compatible with cement mixes.

5. Finding suitable instrumentation for slurries remains a problem.

6. The synthetic slurries tested were more difficult to suspend and keep suspended than the actual sludge will be.

The conclusions have led to a modification of the original operational concept (described in Sect. 1.0). In the modified concept, a large batch (~40,000 gal) of a bentonite suspension will be prepared in an empty Gunit tank. This suspension will be pumped to the jet in the tank being sluiced; the resuspended sludge will be pumped from this tank, discharged into the tank containing the suspension, and mixed with it. Circulation will be continued in this manner until the concentration of resuspended sludge reaches the desired value, at which time the recirculation will be stopped and the batch of resuspended sludge will be pumped to the ILW tanks.

Some potential problems inherent to this concept have not yet been resolved. These problems include:

1. Continuous vision into the tank being sluiced will almost certainly be necessary. We do not know to what extent the generation of fog or spray by the sluicing jet will interfere with this vision.

2. Suitable instrumentation, particularly indicators of viscosity, remains to be developed.

3. The sludge deposits are not homogeneous, and some batch of resuspended sludge may be quite atypical. Such a deviation from the norm should be recognizable quickly enough for corrective action to be taken. Instrumentation to detect abnormal slurry properties will be needed.

APPENDIX

A-1. Radiochemical Analysis of Tanks W-5, W-6, and W-8

The sludge layer in tank W-5 is 9.5 ft deep. Five samples were taken of this sludge in 1975 at various depths. These samples were taken through an opening near the outer wall of the tank. A second set of samples was taken in 1976. Table A-1 gives the reported analytical results; Table A-2 gives the calculated amounts of the various radionuclides in each sludge layer.

The sludge layer in tank W-6 is 3.8 ft deep. Five samples were taken of this sludge in 1975, and a second set of samples was taken in 1976. Table A-3 gives the reported analytical results; Table A-4 gives the calculated quantities of the various radionuclides in each sludge layer.

The sludge layer in tank W-8 is 6.7 ft deep. Five samples were taken of this sludge in 1975, and a second set of samples was taken in 1976. Table A-5 gives the reported analytical results; Table A-6 gives the calculated quantities of the various radionuclides in each sludge layer.

A-2. Chemical Analysis of Tanks W-5, W-6, and W-8

Fractions of the 1975 samples of tanks W-5, W-6, and W-8 were dried, and x-ray analysis data were taken. The results are presented in Tables A-7, A-8, and A-9 as mole percent of elements heavier than sodium (lighter elements cannot be determined by this technique). Sample W-5-1 was observed to contain some lightweight material, which was analyzed separately as W-5-1, top.

Table A-1. Analysis of sludge in tank W-5

	1975					1976				
Distance from bottom, ft	7.7	6.2	5.2	3.2	1.70	7.7	6.2	4.7	3.2	1.7
Density, g/ml	1.17	1.30	1.18	1.50	1.76	1.58	1.79	1.69	2.23	1.67
Solids, %	25.8	12.1	a	42.7	59.6	26.0	22.8	20.3	20.2	46.4
Uranium, mg/g	55.7	138.4	136.0	589.0	122.0	7.8	11.3	12.4	18.3	471.0
Thorium, mg/g	213.0	138.0	38.0	5.9	11.6	65.6	285.9	179.4	218.0	30.8
²³⁹ Pu, cpm/g ^b	0	4.4 x 10 ⁵	2.3 x 10 ⁶	1.4 x 10 ⁷	1.6 x 10 ⁶	7.2 x 10 ⁵	4.3 x 10 ⁵	1.3 x 10 ⁶	5.7 x 10 ⁵	1.5 x 10 ⁷
²³⁸ Pu, cpm/g	5.8 x 10 ⁶	2.6 x 10 ⁶	2.9 x 10 ⁶	2.3 x 10 ⁷	2.2 x 10 ⁶	1.4 x 10 ⁶	4.3 x 10 ⁵	2.6 x 10 ⁸	4 x 10 ⁶	1.9 x 10 ⁷
²⁴⁴ Cm, cpm/g	1.4 x 10 ⁸	1 x 10 ⁷	7.5 x 10 ⁵	2.8 x 10 ⁶	1.1 x 10 ⁶	6.8 x 10 ⁷	8 x 10 ⁷	1.1 x 10 ⁸	1.1 x 10 ⁸	5.5 x 10 ⁶
⁹⁰ Sr, dpm/g ^c	1.0 x 10 ¹⁰	8.7 x 10 ⁹	4.2 x 10 ⁹	3.0 x 10 ⁹	1.2 x 10 ⁹	1.1 x 10 ¹⁰	7.6 x 10 ⁹	9.1 x 10 ⁹	8.7 x 10 ⁹	3.5 x 10 ⁹
⁶⁰ Co, dpm/g	1.8 x 10 ⁸	4.4 x 10 ⁷	8.9 x 10 ⁷	9.4 x 10 ⁶	3.4 x 10 ⁶	5.5 x 10 ⁷	5.2 x 10 ⁷	1.6 x 10 ⁸	1.2 x 10 ⁸	1 x 10 ⁷
¹⁰⁶ Ru, dpm/g	1.2 x 10 ⁸	5.3 x 10 ⁶	7.1 x 10 ⁶	3.0 x 10 ⁶	1.1 x 10 ⁶	1.5 x 10 ⁸	6.1 x 10 ⁷	1.1 x 10 ⁸	9.5 x 10 ⁷	3.2 x 10 ⁶
¹⁵² Eu, dpm/g	5.5 x 10 ⁷	2.2 x 10 ⁶	9.6 x 10 ⁶	1.7 x 10 ⁶	3.4 x 10 ⁵	1.2 x 10 ⁷	3 x 10 ⁷	7.1 x 10 ⁷	3.9 x 10 ⁷	1.8 x 10 ⁶
¹⁵⁴ Eu, dpm/g	9.3 x 10 ⁷	3.1 x 10 ⁶	8.6 x 10 ⁶	6.7 x 10 ⁷	9.7 x 10 ⁶	2.2 x 10 ⁷	4 x 10 ⁷	5.4 x 10 ⁷	5.1 x 10 ⁹	6.2 x 10 ⁷
¹³⁷ Cs, dpm/g	8.1 x 10 ⁷	2.8 x 10 ⁷	1.6 x 10 ⁷	5.2 x 10 ⁷	1.9 x 10 ⁷	3.6 x 10 ⁷	3.9 x 10 ⁷	4.8 x 10 ⁷	4 x 10 ⁷	2.5 x 10 ⁷
¹⁴⁴ Ce, dpm/g	4.6 x 10 ⁷	1.4 x 10 ⁶	2.7 x 10 ⁶	2.6 x 10 ⁶	4.5 x 10 ⁵	1.2 x 10 ⁸	2.4 x 10 ⁷	7.1 x 10 ⁷	4.9 x 10 ⁷	4.6 x 10 ⁶

^aSpilled sample.^bCounts per minute per gram.^cDisintegrations per minute per gram.

Table A-2. Calculated composition of sludge layers, tank W-5

Depth (ft)	Uranium (kg)	Thorium (kg)	²³⁹ Pu (Ci)	²³⁸ Pu (Ci)	²⁴⁴ Cm (Ci)	⁹⁰ Sr (Ci)	⁶⁰ Co (Ci)	¹⁰⁶ Ru (Ci)	¹⁵² Eu (Ci)	¹⁵⁴ Eu (Ci)	¹³⁷ Cs (Ci)	¹⁴⁴ Ce (Ci)
<u>1975</u>												
9.5 to 6.95	2.4×10^3	9.3×10^3		612	4.6×10^3	1.9×10^5	3.5×10^3	2.1×10^3	1.04×10^3	1.8×10^3	1.5×10^3	980
6.95 to 5.7	1.8×10^3	1.9×10^3	14	35	97	5.1×10^4	260	32	13	18	163	8
5.7 to 4.2 ^a												
4.2 to 2.45	3.1×10^4	320	750	970	109	7.1×10^4	225	72	41	1.6×10^3	1.3×10^3	340
2.45 to bottom	1.7×10^4	1.7×10^3	193	238	120	7.5×10^4	22	71	22	620	1.2×10^3	29
<u>1976</u>												
9.5 to 6.95	454	3.8×10^3	36	68	3.4×10^3	2.8×10^5	1.4×10^3	3.8×10^3	310	590	940	3.1×10^3
6.95 to 5.45	384	977	13	13	2.4×10^3	1.2×10^5	800	900	460	620	600	360
5.45 to 3.95	351	5.1×10^3	33	63	2.8×10^3	1.2×10^5	2.1×10^3	1.4×10^3	570	700	620	920
3.95 to 2.45	684	818	19	129	3.4×10^3	1.5×10^5	2.1×10^3	1.6×10^3	650	8.6×10^4	670	820
2.45 to bottom	4.9×10^4	3.2×10^3	1.4×10^3	1.8×10^3	497	1.6×10^5	480	153	90	2.9×10^3	1.2×10^3	220

^aSample spilled; no analysis for percent solids.

Table A-3. Analysis of sludge in tank W-6

	1975					1976				
Distance from bottom, ft	2.8	2.5	1.8	1.5	0.8	4.0	3.0	2.5	12.0	1.0
Density, g/ml	1.30	1.61	1.42	1.38	1.51	1.283	0.764	1.147	1.852	1.418
Solids, %	48.3	37.6	57.8	51.8	49.7	24.2	25.3	29.2	25.4	37.3
Uranium, mg/g	49.3	23.9	2.4	2.0	3.1	4.3	9.2	2.4	4.7	4.9
Thorium, mg/g	<5	18.1	41.5	59.8	8.8	82.0	151.0	79.0	121.0	76.0
²³⁹ Pu, cpm/g ^a	3.3×10^6	2.2×10^6	1.4×10^5	b	b	b	b	b	b	b
²³⁸ Pu, cpm/g	1.7×10^6	5×10^5	b	b	2×10^5	b	b	b	b	b
²⁴⁴ Cm, cpm/g	1.8×10^6	1.2×10^6	1.5×10^6	3.3×10^5	5.2×10^6	7.6×10^7	7.2×10^7	1.2×10^8	9.5×10^7	3.1×10^7
⁹⁰ Sr, dpm/g ^c	2.6×10^9	3.3×10^9	3.1×10^8	1.8×10^8	4.7×10^8	5.1×10^7	2.7×10^9	1.6×10^9	9.1×10^9	3×10^9
⁶⁰ Co, dpm/g	4×10^6	2.9×10^6	1×10^6	2.2×10^5	2.4×10^6	1.1×10^8	1.3×10^8	1×10^8	4.1×10^7	1.4×10^7
¹⁰⁶ Ru, dpm/g	1×10^7	8.2×10^6	4.1×10^6	1.3×10^6	9.3×10^6	3.1×10^8	1.6×10^8	2.4×10^8	1×10^8	2.6×10^7
¹⁵² Eu, dpm/g	3.4×10^6	b	b	b	b	d	d	d	d	d
¹⁵⁴ Eu, dpm/g	3.8×10^6	4.2×10^6	3.4×10^5	b	b	1.4×10^7	1.1×10^7	2.3×10^7	8.8×10^6	2.8×10^6
¹³⁷ Cs, dpm/g	3.1×10^8	2.8×10^8	4.4×10^7	7.2×10^7	3.3×10^7	8.2×10^7	6.4×10^7	1.4×10^8	1.6×10^8	9.2×10^7
¹⁴⁴ Ce, dpm/g	d	d	d	d	d	1.2×10^8	5.9×10^8	1.2×10^8	2.9×10^7	8.9×10^6

^aCounts per minute per gram.^bConcentration below confidence level.^cDisintegrations per minute per gram.^dThis radionuclide not reported in this analysis.

Table A-4. Calculated composition of sludge layers, tank W-6

Depth (ft)	Uranium (kg)	Thorium (kg)	²³⁹ Pu (Ci)	²³⁸ Pu (Ci)	²⁴⁴ Cm (Ci)	⁹⁰ Sr (Ci)	⁶⁰ Co (Ci)	¹⁰⁶ Ru (Ci)	¹⁵² Eu (Ci)	¹⁵⁴ Eu (Ci)	¹³⁷ Cs (Ci)	¹⁴⁴ Ce (Ci)
<u>1975</u>												
3.8 to 2.65	2 x 10 ³	200	114	59	63	4.7 x 10 ⁴	72	179	61	68	5.6 x 10 ³	a
2.65 to 2.15	4 x 10 ³	300	32	7	18	4.8 x 10 ⁴	22	62	0	32	2.1 x 10 ³	a
2.15 to 1.65	55	950	2.8	0	30	3.2 x 10 ³	10	42	0	3.5	470	a
1.65 to 1.15	40	1.2 x 10 ³	0	0	6	1.6 x 10 ³	20	11.5	0	0	650	a
1.15 to bottom	4.5 x 10 ³	420	0	8	216	100	51	202	0	0	720	a
<u>1976</u>												
4 to 3.5	37	706	0	0	565	796	420	1.2 x 10 ³	a	56	319	450
3.5 to 2.75	74	1.2 x 10 ³	0	0	500	9.7 x 10 ³	465	120	a	41	233	2.2 x 10 ³
2.75 to 2.0	33	1.1 x 10 ³	0	0	1.5 x 10 ³	9.7 x 10 ³	653	1.5 x 10 ³	a	142	890	773
2.0 to 0.5	185	4.7 x 10 ³	0	0	3.2 x 10 ³	1.6 x 10 ⁵	729	1.8 x 10 ³	a	155	2.8 x 10 ³	503
0.5 to bottom	72	1.1 x 10 ³	0	0	400	2 x 10 ⁴	93	173	a	19	607	59

^aThis radionuclide not reported in this analysis.

Table A-5. Analysis of sludge in tank W-8

1975						1976				
Distance from bottom, ft	5.7	4.7	3.7	2.7	1.7	6.7	5.7	4.7	3.7	2.7
Density, g/ml	2.37	1.28	1.32	1.28	1.22	1.957	1.511	2.463	2.197	1.565
Solids, %	16.8	49.0	30.1	37.3	27.5	33.3	30.5	38.2	30.1	36.9
Uranium, mg/g	27.2	70.3	9.33	22.8	36.3	19.3	14.0	9.1	16.2	54.0
Thorium, mg/g	221.5	143.6	116.1	152.8	95.7	179.0	274.0	120.0	68.0	46.0
²³⁹ Pu, cpm/g ^a	b	b	b	b	6.7 x 10 ⁶	b	b	7.2 x 10 ⁵	1.9 x 10 ⁶	1.6 x 10 ⁶
²³⁸ Pu, cpm/g	b	b	3.4 x 10 ⁷	3 x 10 ⁷	1.2 x 10 ⁷	b	b	6.5 x 10 ⁶	7.7 x 10 ⁶	4.8 x 10 ⁶
²⁴⁴ Cm, cpm/g	5.8 x 10 ⁷	1.8 x 10 ⁸	8.4 x 10 ⁷	9.4 x 10 ⁷	3.6 x 10 ⁷	2 x 10 ⁷	4.9 x 10 ⁷	1.9 x 10 ⁷	1.1 x 10 ⁷	4.7 x 10 ⁶
⁹⁰ Sr, dpm/g ^c	1.1 x 10 ¹⁰	9.8 x 10 ⁹	3.2 x 10 ¹⁰	1.4 x 10 ¹⁰	9.3 x 10 ⁹	2.2 x 10 ⁹	3.5 x 10 ⁹	2.6 x 10 ⁹	5.1 x 10 ⁹	5 x 10 ⁹
⁶⁰ Co, dpm/g	4.3 x 10 ⁷	4.9 x 10 ⁷	7.5 x 10 ⁷	7.5 x 10 ⁷	2.8 x 10 ⁷	2.9 x 10 ⁷	1.6 x 10 ⁷	1 x 10 ⁷	1.8 x 10 ⁷	9.2 x 10 ⁶
¹⁰⁶ Ru, dpm/g	1.5 x 10 ⁷	5.8 x 10 ⁷	2.4 x 10 ⁷	5.1 x 10 ⁷	1.5 x 10 ⁷	3.2 x 10 ⁷	1.3 x 10 ⁷	9 x 10 ⁶	1.3 x 10 ⁷	1.5 x 10 ⁷
¹⁵² Eu, dpm/g	2.7 x 10 ⁷	1.9 x 10 ⁸	4.0 x 10 ⁷	6.4 x 10 ⁷	2.9 x 10 ⁷	d	d	d	d	d
¹⁵⁴ Eu, dpm/g	4.6 x 10 ⁷	4.2 x 10 ⁸	5.9 x 10 ⁷	1.2 x 10 ⁸	5.1 x 10 ⁷	1.4 x 10 ⁷	9.8 x 10 ⁶	1.3 x 10 ⁷	1.4 x 10 ⁷	8.3 x 10 ⁶
¹³⁷ Cs, dpm/g	2.5 x 10 ⁸	3.8 x 10 ⁸	4.1 x 10 ⁸	3.7 x 10 ⁸	3.8 x 10 ⁸	2.5 x 10 ⁸	8 x 10 ⁷	4.4 x 10 ⁸	6.3 x 10 ⁸	6.4 x 10 ⁸
¹⁴⁴ Ce, dpm/g	d	d	d	d	d	8.4 x 10 ⁶	4.2 x 10 ⁶	1.4 x 10 ⁷	7.8 x 10 ⁶	7.8 x 10 ⁶

^aCounts per minute per gram.^bConcentration below confidence level.^cDisintegrations per minute per gram.^dThis radionuclide not reported in this analysis.

Table A-6. Calculated composition of sludge layers, tank W-8

Depth (ft)	Uranium (kg)	Thorium (kg)	²³⁹ Pu (Ci)	²³⁸ Pu (Ci)	²⁴⁴ Cm (Ci)	⁹⁰ Sr (Ci)	⁶⁰ Co (Ci)	¹⁰⁶ Ru (Ci)	¹⁵² Eu (Ci)	¹⁵⁴ Eu (Ci)	¹³⁷ Cs (Ci)	¹⁴⁴ Ce (Ci)
<u>1975</u>												
6.7 to 5.2	522	4.2 x 10 ³	0	0	950	8.9 x 10 ⁴	380	130	229	390	2.3 x 10 ³	a
5.2 to 4.2	2.5 x 10 ³	5 x 10 ³	0	0	5.5 x 10 ³	1.5 x 10 ⁵	770	900	3 x 10 ³	6.6 x 10 ³	6 x 10 ³	a
4.2 to 3.2	206	2.6 x 10 ³	0	641	1.6 x 10 ³	3.2 x 10 ⁵	750	240	400	590	4 x 10 ³	a
3.2 to 2.2	605	4 x 10 ³	0	239	930	6 x 10 ⁵	890	640	800	1.5 x 10 ³	4.5 x 10 ³	a
2.2 to bottom	1.8 x 10 ³	3.9 x 10 ³	231	407	1.3 x 10 ³	1.7 x 10 ⁵	530	280	540	950	7 x 10 ³	a
<u>1976</u>												
6.7 to 6.2	333	3,330	0	0	312	1.8 x 10 ⁴	230	260	a	109	2 x 10 ³	68
6.2 to 5.2	355	7,200	0	0	1.1 x 10 ³	4 x 10 ⁴	178	147	a	113	920	48
5.2 to 4.2	467	6,120	32.5	295	860	6 x 10 ⁴	240	211	a	295	1 x 10 ⁴	339
4.2 to 3.2	611	2,500	60.7	243	340	6 x 10 ⁴	239	217	a	198	9.9 x 10 ³	129
3.2 to bottom	550	4,800	143	426	426	2.3 x 10 ⁴	429	694	a	386	3 x 10 ⁴	362

^aThis radionuclide not reported in this analysis.

Table A-7. X-ray analysis of samples from tank W-5
(mole percent of elements heavier than sodium)

	W5-1 Top	W5-1 Bottom	W5-2	W5-3	W5-4	W5-5
Na						
Al	38.5	6.7	21.4	10.9	12.7	3.4
Si		1.2				
P		0.2	3.2	25.9	0.8	18.6
S	47.6	0.9				
Cl		2.0				
K						
Ca	6.3	15.5	22.9	16.3		59.9
Ti						
Cr			0.6			
Mn		1.4	3.3	1.7	1.0	
Fe		5.2	14.5	4.9	3.9	2.8
Ni			0.2			
Cr		1.6	4.1		2.7	2.2
Zn						
Pb	7.5		16.1	2.9	5.4	
Th		65.5		37.5		
U			13.9		48.1	11.3

Table A-8. X-ray analysis of samples from tank W-6
(mole percent of elements heavier than sodium)

	W6-1	W6-2	W6-3	W6-4	W6-5
Na					9.3
Al	9.5	59.5	46.8	19.9	30.8
Si					
P	2.7				
S	2.2	1.6	0.3		
Cl	2.3	9.5	8.4	5.1	46.5
K				1.0	0.5
Ca	30.6	3.5	2.7	16.1	0.7
Ti					0.04
Cr					
Mn					
Fe	29.6	25.9	38.6	57.9	11.5
Ni	2.9		1.3		
Cr	4.2		1.8		0.7
Zn	2.0				
Pb	10.8				
Th					
U	3.2				

Table A-9. X-ray analysis of samples from tank W-8
(mole percent of elements heavier than sodium)

	W8-1	W8-2	W8-4	W8-5
Na				
Al	9.5	10.7	4.1	22.6
Si				
P	1.0			0.6
S		6.3	25.9	5.9
Cl	44.0	0.7	5.8	0.2
K				
Ca	5.5	5.0	64.2	13.7
Ti				
Cr				
Mn				
Fe	6.1	2.4		7.6
Ni				
Cr				3.0
Zn				
Pb				
Th	33.9	74.9		38.1
U				8.3

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