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Radioative Waste

STATUS REPORT ON WASTE DISPOSAL IN NATURAL SALT FORMATIONS: II

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Health Physics Division

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ABSTRACT

Radioactive liquid wastes can be stored in cavities in natural salt formations if the structural properties of salt are not adversely affected by chemical interaction, pressure, temperature, and radiation. Analytical studies show that it is possible to store two-year-old waste in a 10-foot-diameter sphere without exceeding a temperature of 200° F. Laboratory tests show that the structural properties of rock salt are not greatly altered by high radiation doses, although high temperatures increase the creep rate for both irradiated and unirradiated samples. Chemical interaction of liquid wastes with salt produces chlorine and other chlorine compound gases, but the volumes are not excessive. The migration of nuclides through the salt and deformation of the cavity can only be studied in the field in undisturbed salt in place. The design of a field experiment is complete, and the materials for construction of the cavity cover and off-gas system have been chosen.

INTRODUCTION

The first status report[1] on radioactive waste disposal in cavities in in natural salt formations characterized the limits of the problem as "the degree to which the desired structural properties of salt are affected by liquid wastes, pressure, temperature, and radiation..." This status report covers some of the theoretical considerations and preliminary laboratory investigations of these effects. The theoretical studies show that the temperature rise of radioactive wastes stored in salt cavities can be kept below acceptable limits by controlling the age of the wastes and the size of the cavity. Laboratory work on chemical compatibility shows that present-day wastes are essentially inert toward rock salt. Additionally, radiation has little effect on the structural properties of rock salt. However, all of the information necessary for the design of the field-scale radioactive experiment cannot be obtained theoretically nor in laboratory tests, because theoretical solutions require a number of simplifying assumptions, and laboratory tests of geological materials are not conclusive, because the samples are removed from their environment and, hence, disturbed. Preliminary field tests will provide information on the effects of heat and chemical interaction on the structural integrity of the cavity, the migration of nuclides from the cavity out into the surrounding salt, and the migration of the cavity itself.

THERMAL CALCULATIONS

The plasticity and structural integrity of cavities in rock salt are dependent upon temperature and pressure. Pressure is a function of the stratigraphy, the percentage of salt excavated, and the configuration of the excavation.

Temperature in a cavity is a function of its depth, size, shape, the rate of heat production of the stored material, and thermal conduction in the surrounding salt. The heat production rate, approximated from Fig. 1, is given as a function of decay time, assuming 800 gal of waste per metric ton of uranium, irradiated for 10,000 Mwd/ton at 33 Mw/ton specific power.[2]

If a limit to the temperature rise within a cavity and the space about it is predetermined, then the time-space-temperature distribution for various sizes, shapes, and heat production rates of the wastes stored within the cavity can be calculated.

Birch has stated "that heating the liquid some hundred degrees (centigrade) or so would not produce drastic physical changes, and that plastic deformation of shale or evaporite layers, if sufficiently thick might continue to provide a tight container."[3] We, therefore, set a 200° F rise as the maximum permissible temperature rise in a salt cavity.

The possible sizes and shapes of cavities and the heat production rates of wastes are so numerous that a few simplifying assumptions were necessary:

(1) Only spherical shapes would be investigated so that an analytical solution would be possible.

(2) Diameters of the spheres would be limited to 10 ft and 20 ft, since it had been shown previously[4] that larger spheres, containing wastes generating heat at a rate of 2 BTU/hr/gal, would exceed the permissible limit of 200° F.

(3) Wastes considered would be limited to those described by Fig. 1 and cooled for 1, 6, and 11 years.

(4) Waste-filled cavities would be located in an infinite body of salt.

(5) Thermal conductivity, density, and specific heat of the salt and the wastes would be constant and independent of changes in temperature.

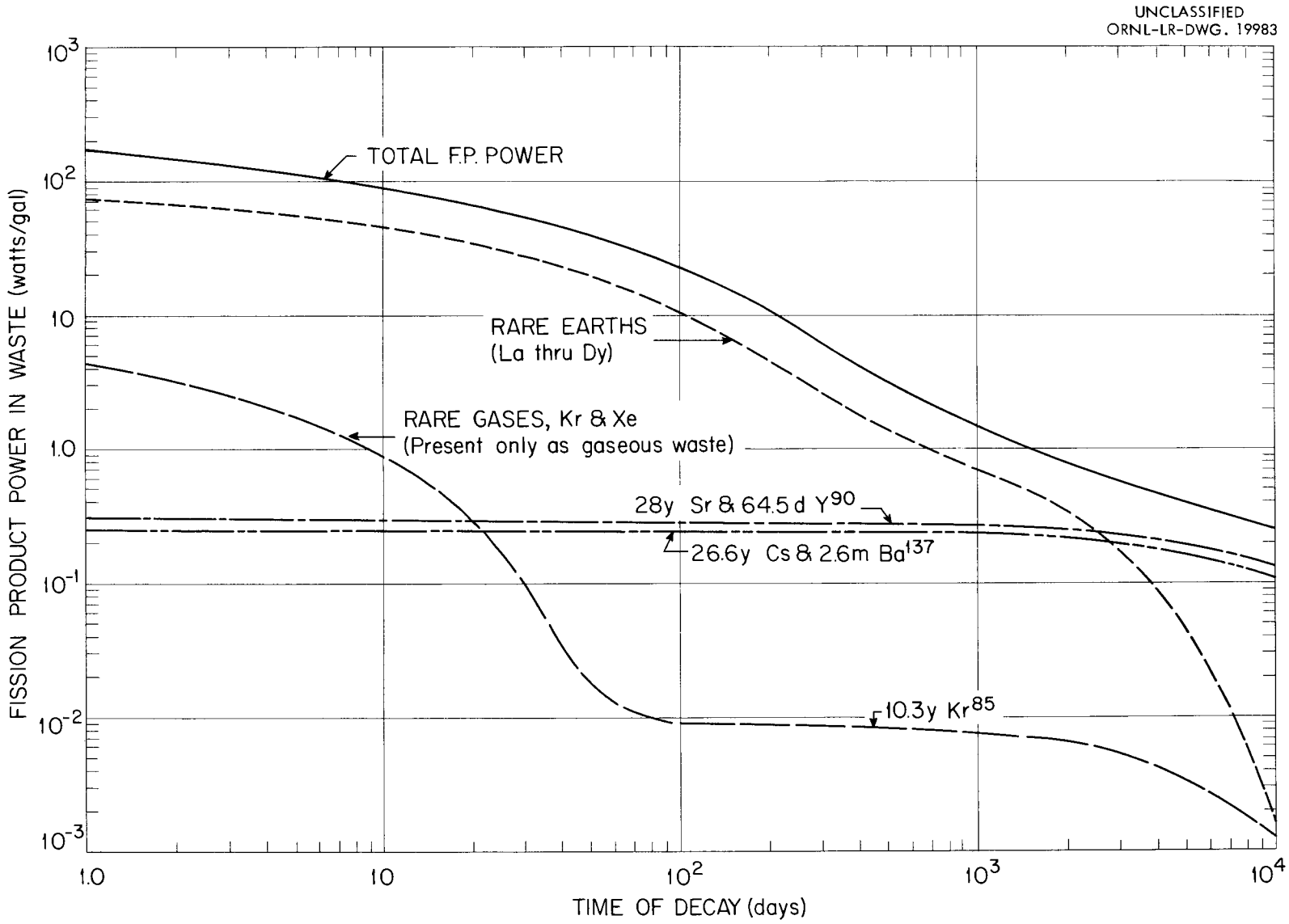

FIG. 1. FISSION PRODUCT POWER IN WASTE AS A FUNCTION OF DECAY TIME BASIS: 800 GAL WASTE PER METRIC TON U, IRRADIATED 10,000 Mwd/ton At 33 Mw/ton SPECIFIC POWER.

(6) Heat would be lost by conduction only.

(7) Phase changes would not occur in the wastes or in the salt.

(8) Convective currents would not develop in the wastes.

(9) All beta and gamma energy would be absorbed in the wastes.

All assumptions, except the extent of salt, are on the safe side; that is, the temperatures calculated would be higher than those actually achieved. The absence of convective currents in the wastes would produce a temperature gradient from the center of the cavity out to the interface of the salt and the waste. If convective currents were present, the temperature would be uniform throughout the sphere. For cavities of the size considered, however, the edge temperature in the conduction case would approach the uniform temperature achieved if convective currents were present.

Statement of Problem

A mathematical statement of the spherical problem is:

$$\frac{1}{\alpha_1}\frac{\partial T_1}{\partial t} = \frac{\partial^2 T_1}{\partial r^2} + \frac{2}{r}\frac{\partial T_1}{\partial r} + \frac{Q(t)}{K_1}, \qquad 0 \leq r < a,\ t > 0, \quad (1)$$

and

$$\frac{1}{\alpha_2}\frac{\partial T_2}{\partial t} = \frac{\partial^2 T_2}{\partial r^2} + \frac{2}{r}\frac{\partial T_2}{\partial r}, \qquad r > a,\ t > 0, \quad (2)$$

where subscripts 1 and 2 refer to the heated sphere and to the infinite body, respectively; T is temperature; t is time; r is radial distance from the center

of the sphere; a is the radius of the sphere; K is thermal conductivity; α is thermal diffusivity; and Q(t) is heat generation per unit time per unit volume.

Initially, the wastes and salt are considered to be at zero temperature. Boundary conditions are such that the temperature and heat flux are continuous at the interface, and the temperature remains finite both at the center of the sphere and at infinity. Then, by the use of La Place Transforms and Duhamel's theorem the following equations were obtained. The temperature inside the cavity is

$$T_1(t) = \int_0^t \sum_{i=1}^{N} A_i \frac{e^{-\lambda_i(t-\tau)}}{2C_2} \left\{ \operatorname{erf}\left[\frac{a-r}{2\sqrt{\alpha_2\tau}}\right] + \operatorname{erf}\left[\frac{a+r}{2\sqrt{\alpha_2\tau}}\right] - \right.$$

$$\left. \sqrt{\frac{4\alpha_2\tau}{r^2\pi}} \left[e^{-(a-r)^2/4\alpha_2\tau} - e^{-(a+r)^2/4\alpha_2\tau} \right] \right\} d\tau,$$

and outside of the cavity

$$T_2(t) = \int_0^t \sum_{i=1}^{N} A_i \frac{e^{-\lambda_i(t-\tau)}}{2C_2} \left\{ \operatorname{erf}\left[\frac{r+a}{2\sqrt{\alpha_2\tau}}\right] - \operatorname{erf}\left[\frac{r-a}{2\sqrt{\alpha_2\tau}}\right] - \right.$$

$$\left. \sqrt{\frac{4\alpha_2\tau}{r^2\pi}} \left[e^{-(r+a)^2/4\alpha_2\tau} - e^{-(r-a)^2/4\alpha_2\tau} \right] \right\} d\tau,$$ where N is the number of fission products. A_i is the heat generation per unit time per unit volume for ith fission product, and λ_i is the disintegration constant of the ith fission product as $\sum_{i=1}^{N} A_i e^{-\lambda_i t}$ is substituted for Q(t). C is the specific heat per unit volume ($C = \frac{K}{\alpha}$); τ is a variable of integration; and erf is the error

function. For rock salt, K_2 equals 2.07 $\frac{\text{BTU}}{\text{ft-hr-}^{\circ}\text{F}}$; α equals 0.07 $\frac{\text{ft}^3}{\text{hr}}$; and C_2 equals 29.7 $\frac{\text{BTU}}{\text{ft}^3\text{-}^{\circ}\text{F}}$.

Summing the integrals of the decay curves of all fission products contributing to heat generation would be arduous. Therefore, the total fission product power, as shown on Fig. 1, is approximated by six exponentials: The decay curves for Cs^{137} and Ba^{137}, Sr^{90} and Y^{90}, long-lived components of the rare earths, short-lived components of the rare earths, long-lived components, and short-lived components of the remainder of the fission products.

Results

Equations for the temperatures both inside and outside the cavity were numerically integrated on the IBM 704 using Simpson's Rule and the Fortran coding system to calculate the temperature rises.[5] Curves of these data are shown in Figs. 2 to 7.

For the 10-foot-diameter sphere and one-year-old waste (Fig. 2), it is seen that the maximum rise in temperature at the center of the sphere is 530° F at 1000 hr and 315° F at the surface of the sphere at 1500 hr. In Fig. 3 the maximum temperature rise for six-year-old waste at the center of the sphere is 100° F at 4500 hr and 65° at the surface at 7000 hr. In Fig. 4 the maximum temperature rise at the center of the sphere of eleven-year-old waste is 65° F at 7000 hr and 40° F at the surface at 10,000 hr. As interpolated from these data, two-year-old wastes stored in a 10-foot-diameter sphere would reach a peak temperature of 200° F in less than five months.

For the 20-foot-diameter sphere (Figs. 5, 6, and 7), it is seen that the same wastes could not be stored in cavities of this size without boiling.

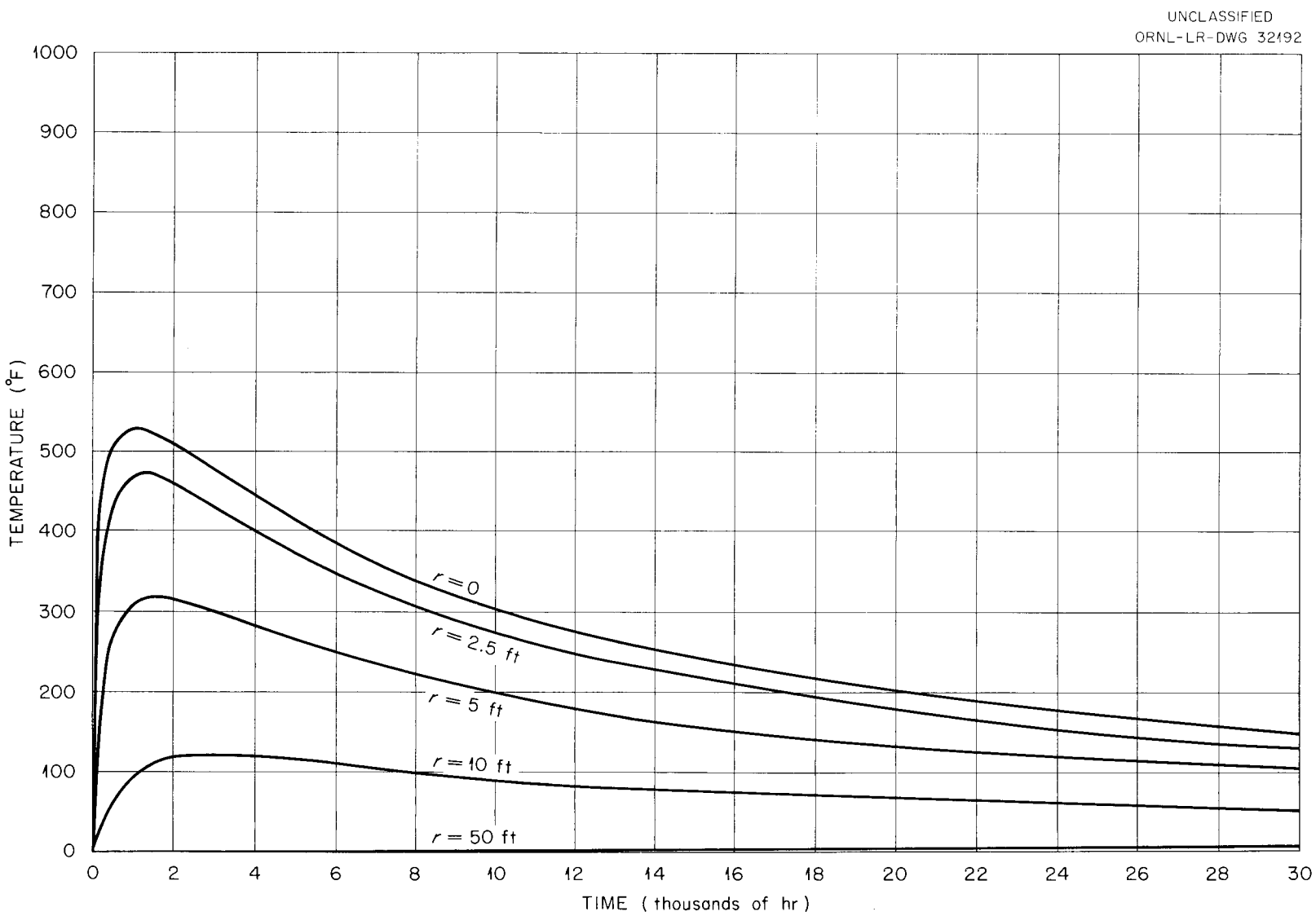

Fig. 2. 1 Year Old Waste – Sphere of 5-ft Radius, Surrounded by an Infinite Bed of Salt.

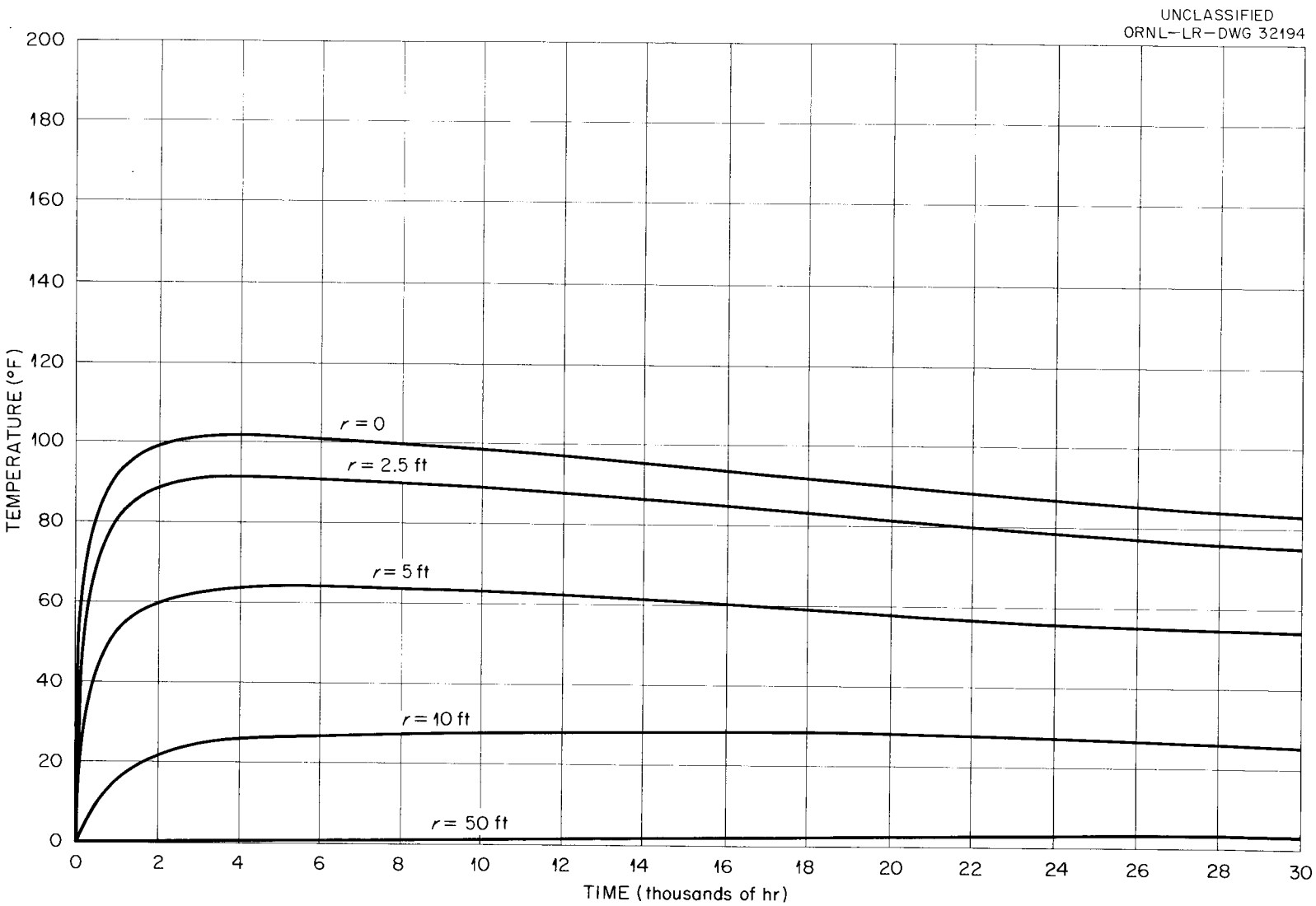

Fig. 3. 6 Year Old Waste – Sphere of 5-ft Radius. Surrounded by an Infinite Bed of Salt.

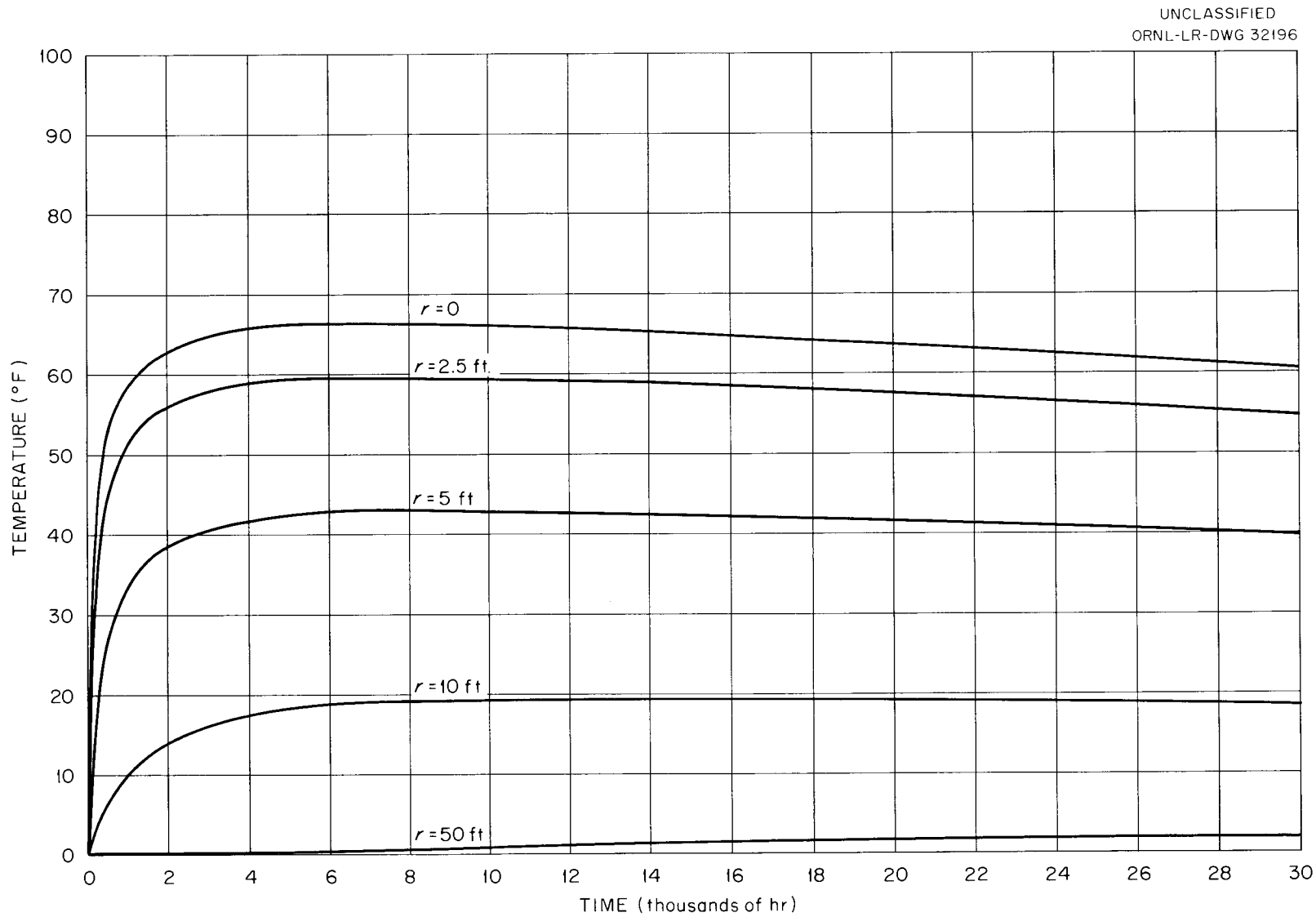

Fig. 4. 11 Year Old Waste-Sphere of 5-ft Radius. Surrounded by an Infinite Bed of Salt.

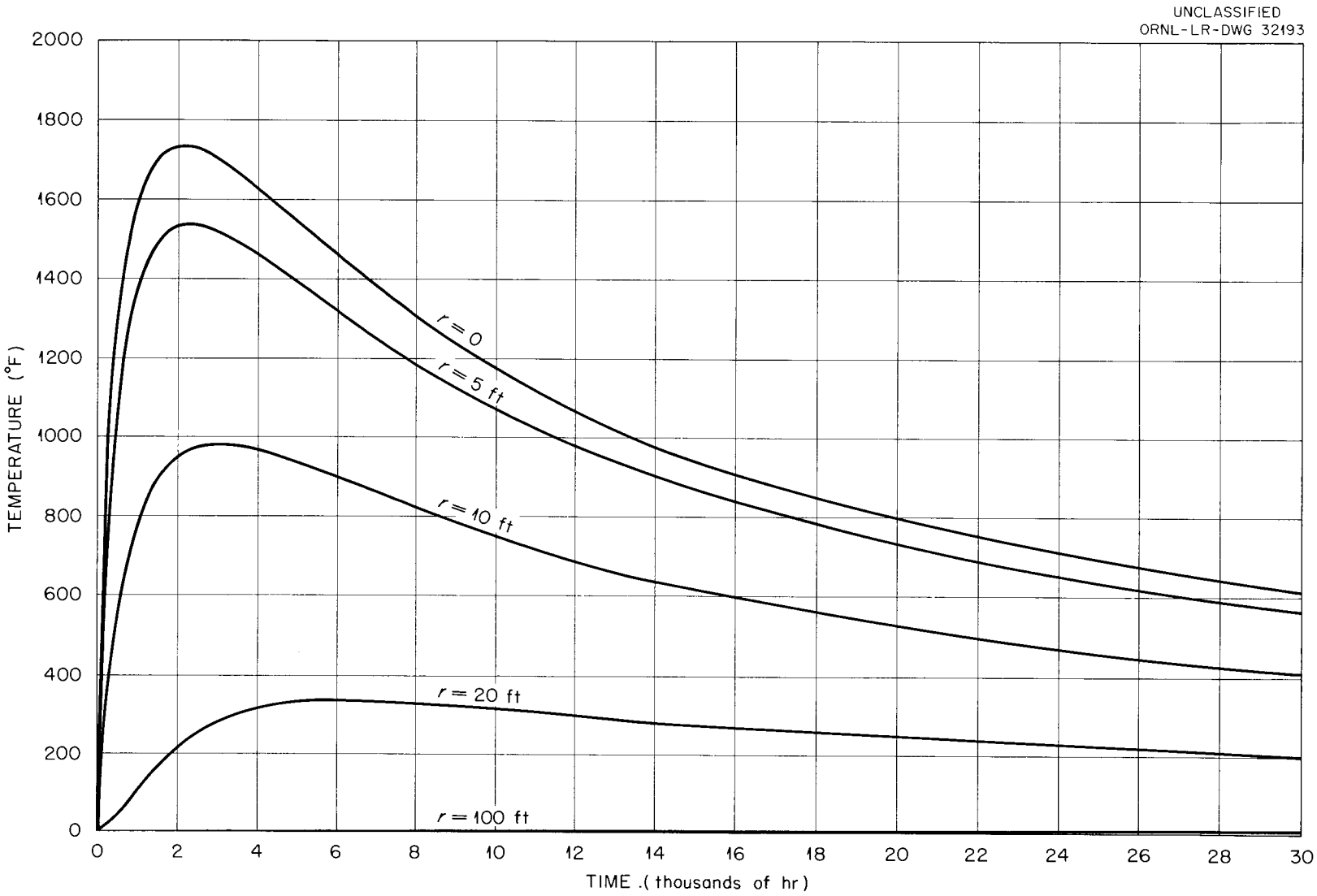

Fig. 5. 1 Year Old Waste — Sphere of 10-ft Radius, Surrounded by an Infinite Bed of Salt.

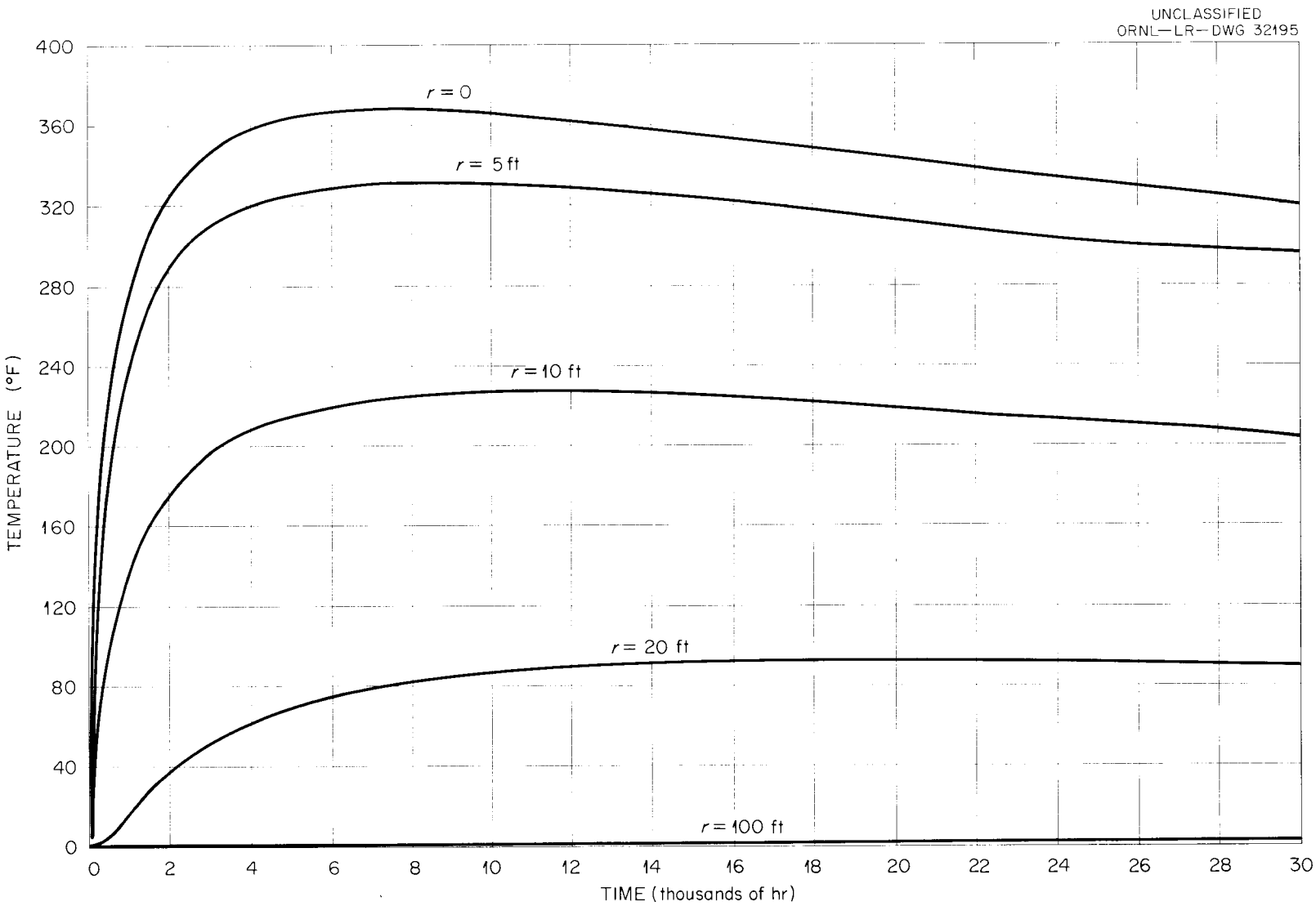

Fig. 6. 6 Year Old Waste–Sphere of 10-ft Radius. Surrounded by an Infinite Bed of Salt.

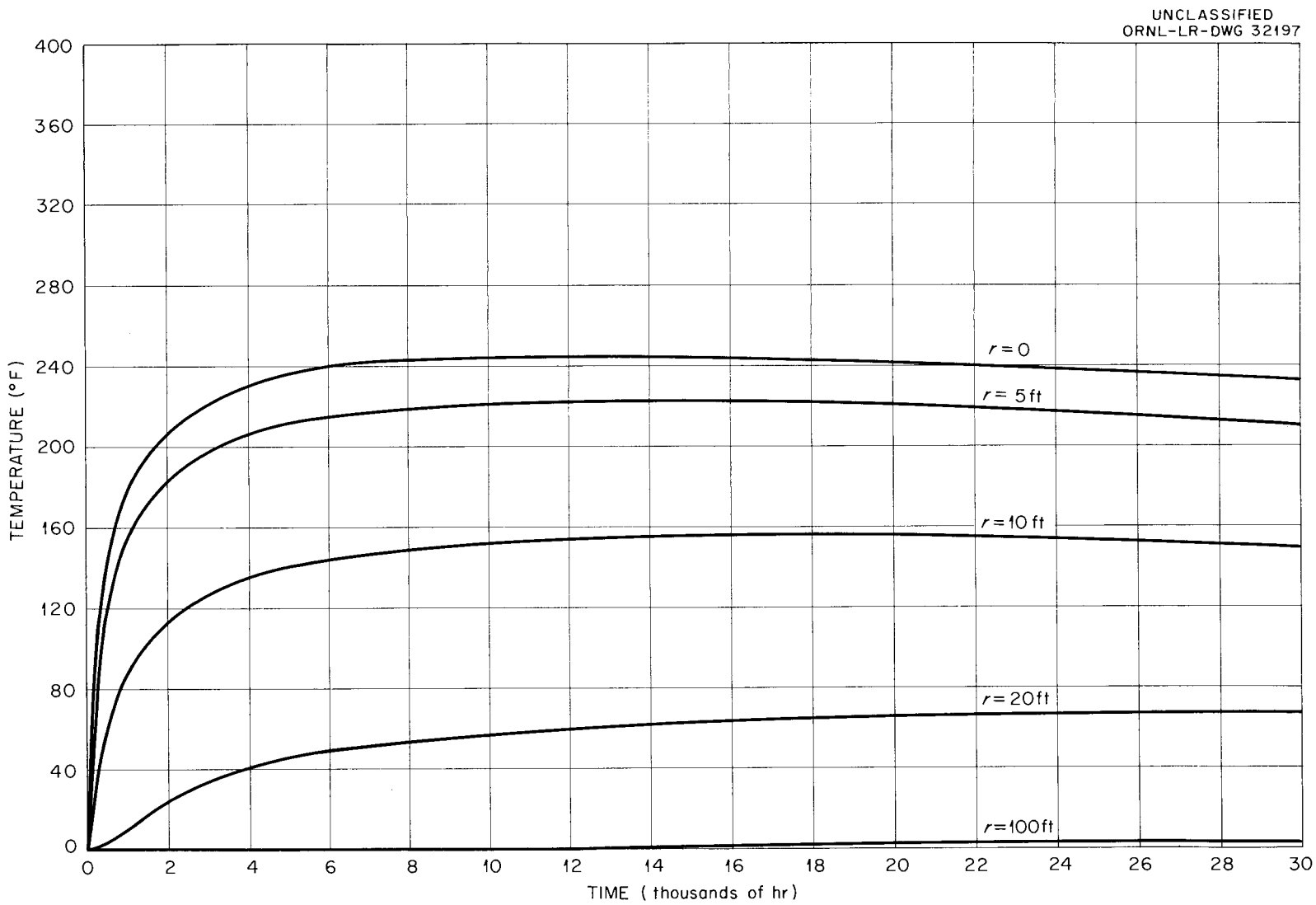

Fig. 7. 11 Year Old Waste – Sphere of 10 – ft Radius. Surrounded by an Infinite Bed of Salt.

These results are substantially the same as the calculations of Birch[6] and Schecter.[7] They considered only temperature-time distributions for the conduction case; in addition, they considered the convection case in their calculations. For a sphere of 5-foot radius, Schecter found a drop of 35% from center-of-the-sphere temperature in the conduction case to uniform sphere temperature in the convection case. Our calculations for spheres of this size show a 40% reduction from center temperature to surface temperature in the conduction case, or little difference between surface temperature in the conduction case and sphere temperature in the convection case.

Therefore, we may conclude that the thermal problem in salt disposal will cause no serious difficulties if the size of the cavities and the age and dilution of the wastes are controlled.

STRUCTURAL PROPERTIES OF ROCK SALT

The rock salt surrounding waste-filled cavities will be subjected to certain stresses induced by elevated temperatures and pressures, and radiation. The response of crystal aggregates of rock salt to temperature, pressure, and radiation was investigated to determine the stress-strain relationships and the creep rate.[8] Stress-strain relationships were used to determine the compressive strength, modulus of elasticity, yield strength (0.2% offset), and apparent elastic limit.

Experimental Procedure

The American Society of Testing Materials does not have a standard procedure for the testing of rock-salt aggregates. Consequently, the test procedures for wood and stone were investigated and the more pertinent provisions

applied to the testing of rock salt.[9] Two-inch cubes of salt, randomly selected, were used as test specimens. Specimens of dome salt were machined from a large block taken from the Morton Salt Company's mine at Grand Saline, Texas. Specimens of bedded-deposit salt were machined from a block taken from the Carey Salt Company's mine at Hutchinson, Kansas. The purity of dome salt was greater than 98% NaCl and that of the bedded salt was 95% NaCl.

Machined specimens were irradiated in the Cobalt Storage Facility of the Operations Division at ORNL. Approximately 200,000 curies of Co^{60} in aluminum clad cylinders are stored about a one cubic foot chamber below ground level to produce an exposure dose rate of 3.2×10^6 roentgens per hour, as measured with a ceric sulfate chemical dosimeter.

Specimens were tested with the assistance of the Mechanical Testing Section of the Metallurgy Division at ORNL. A 120,000-lb capacity Baldwin-Tate-Emery Universal testing machine was used for load application. Deformation was measured by a Bell-Crank-Anvil extensometer. This apparatus automatically plotted a total load versus total deformation curve which was then reduced to a stress-strain diagram.

Tests at Room Temperature

Stress-Strain Relationships of Unirradiated Bedded and Dome Salt.-- Three specimens each of dome salt, bedded salt loaded parallel to the planes of stratification, and bedded salt loaded perpendicular to the planes of stratification were tested in order to establish a control with which to compare irradiated specimens. The results of these tests are shown graphically in Fig. 8 and in tabular form in Table 1.

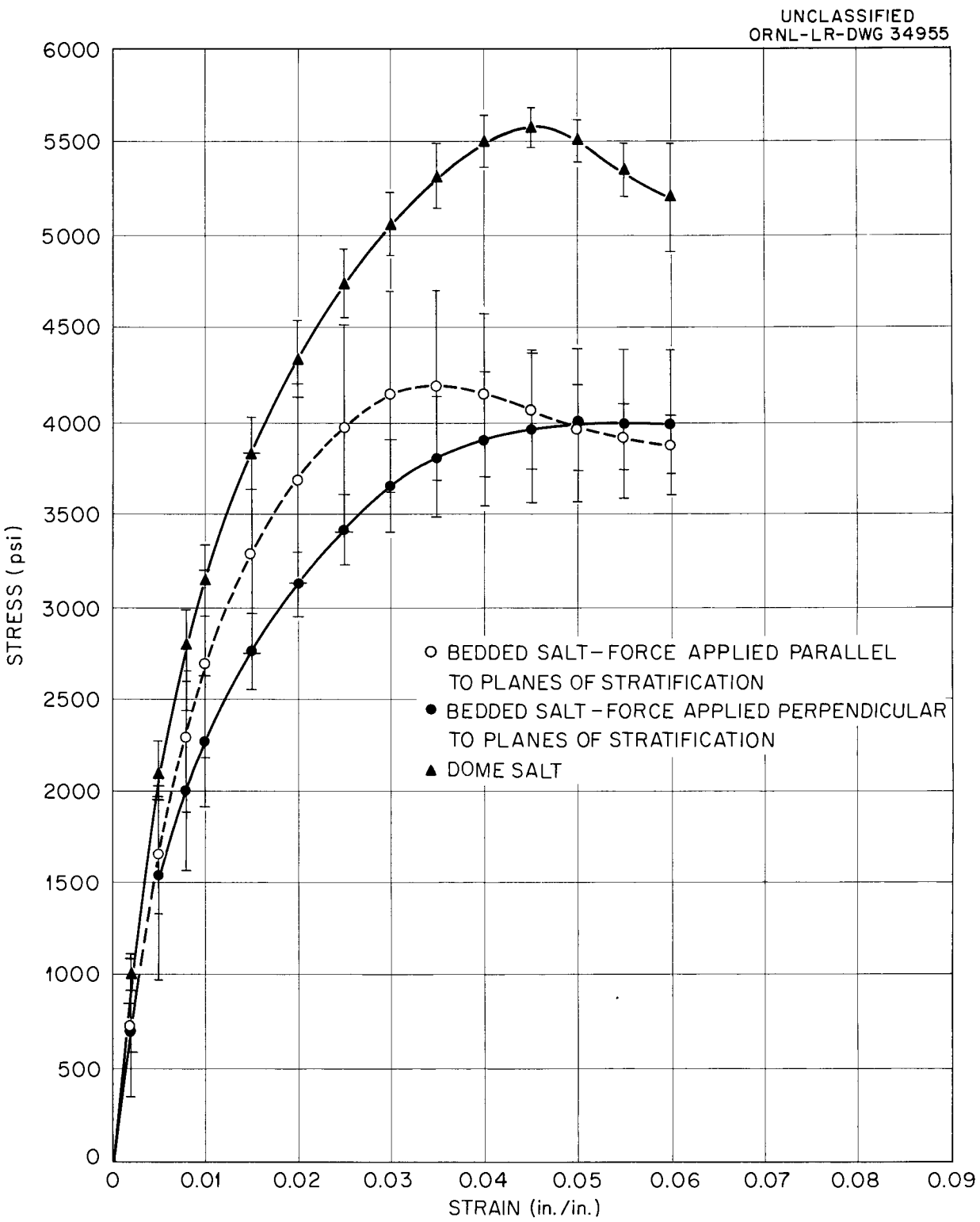

Fig. 8. Compression Test of Unirradiated Salt at Room Temperature.

Table 1. Structural Properties of Rock Salt

Type of Salt	Temperature	Exposure Dose(r)	Compressive Strength (psi)	Yield Strength (psi)	Apparent Elastic Limit (psi)	Modulus of Elasticity (psi) x 10^6
Dome	Room	0	5600 ± 100[a]	2900 ± 150	2000 ± 100	.50 ± .05
Bedded Parallel*	Room	0	4200 ± 500	2500 ± 300	1900 ± 300	.35 ± .06
Bedded Perpendicular**	Room	0	4000 ± 400	2300 ± 300	1700 ± 200	.36 ± .17
Bedded Parallel	Room	0	4200 ± 500	2500 ± 300	1900 ± 300	.35 ± .06
Bedded Parallel	Room	10^6	4500 ± 500	2500 ± 150	1900 ± 200	.32 ± .06
Bedded Parallel	Room	10^7	4600 ± 400	2700 ± 250	2100 ± 200	.35 ± .02
Bedded Parallel	Room	10^8	4600 ± 150	3400 ± 250	2800 ± 200	.36 ± .09
Bedded Parallel	Room	5×10^8	3500 ± 50	2700 ± 200	2000 ± 300	.39 ± .07
Bedded Parallel	200° C	0	3900 ± 200	1600 ± 200	1400 ± 100	.090 ± .01
Bedded Parallel	200° C	5×10^8	3400 ± 400	1900 ± 250	1800 ± 250	.12 ± .02

* Bedded parallel - bedded salt, force applied parallel to the planes of stratification.
** Bedded perpendicular - bedded salt, force applied perpendicular to the planes of stratification.
a - All results have been rounded off to the nearest 100 psi and the standard deviation to the nearest 50 psi.

Dome salt has greater compressive and yield strengths than bedded salt. Previous work with synthetic crystals indicated that small amounts of impurities added to sodium chloride increased the tensile strength of salt. The results for synthetic and single crystals are not applicable to aggregates of crystals.[10] The binding strength between crystals becomes an important factor in the strength of the aggregates. The presence of other substances in the specimens, anhydrites and shales, is at least partially responsible for the greater variation exhibited by the bedded salt. Dome salt, although neither isotropic nor homogeneous, is much more uniform with respect to impurities than bedded salt. The mined block of bedded salt was interstratified with anhydrite and shale bands of various thicknesses.

Bedded salt is slightly stronger when loaded parallel to planes of stratification than when loaded perpendicularly. Although this difference is within the statistical variation of the experiment, the same trend is evident in creep tests.

Creep Tests - Unirradiated Bedded and Dome Salt.-- The creep rate resulting from the application of a constant load of 2500 pounds per square inch for 100 minutes was measured. The creep rates of three types of salt are shown in Fig. 9. After an initial rise, due mostly to elastic compression, the strain increases very slowly with time. As in the stress-strain tests, dome salt is strongest (least creep under the same load conditions) and exhibits the least variation from sample to sample. Bedded salt loaded parallel to the planes of stratification is again stronger than bedded salt load perpendicular to the planes of stratification, though still within the statistical variation of the experiment.

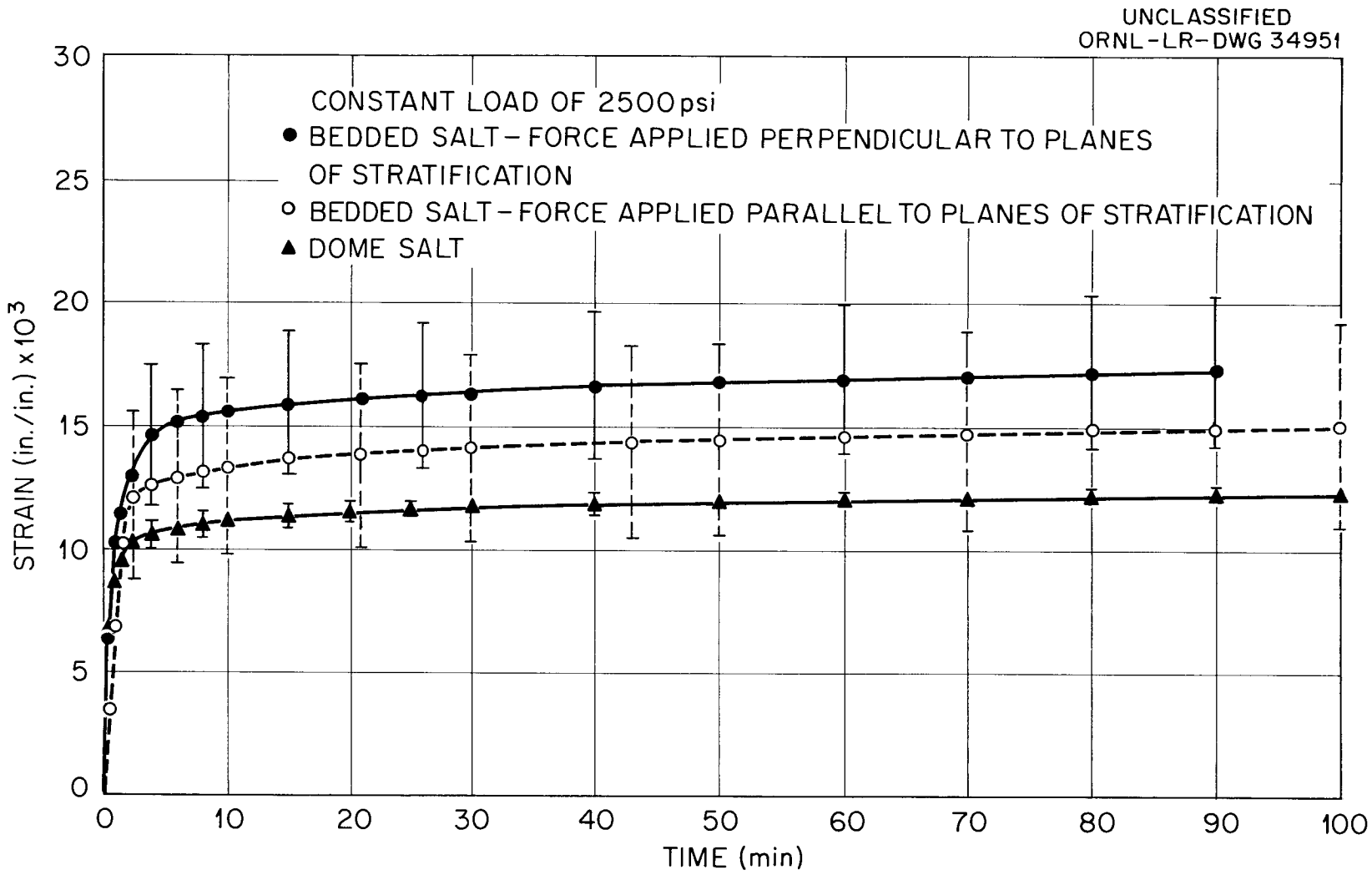

Fig. 9. Creep Test of Unirradiated Salt at Room Temperature.

Stress-Strain Relationship of Irradiated Bedded Salt Loaded Parallel to the Planes of Stratification.-- Bedded salt samples were irradiated for periods to give exposure doses of 10^6, 10^7, 10^8, and 5×10^8 roentgens, assuming a dose rate constant with time.

The stress-strain curves of irradiated bedded salt are shown in Fig. 10, and the data are tabulated in Table 1. From Fig. 10 it is seen that there is very little difference in the stress-strain relationship up to a stress of 2000 pounds per square inch. The compressive strength increases with exposures up to 5×10^8 r, where a marked decrease is observed. The compressive strengths up to exposures of 5×10^8 r are within the statistical variation of the experiment, while the low value obtained at 5×10^8 is not.

The expected increase in the modulus of elasticity is observed only at 5×10^8 r and is within the limit of statistical variation. In fact, at 10^6 r the modulus of elasticity, yield strength, and apparent elastic limit are slightly lower than for the unirradiated specimens.

The yield strength and apparent elastic limit reach a maximum at 10^8 r. However, at 5×10^8 r the yield strength and apparent elastic limit, unlike the compressive strength, are greater than for the unirradiated specimens.

Creep Rate of Irradiated Bedded Salt Loaded Parallel to the Planes of Stratification.-- The effect of radiation on the rate of creep was investigated by testing three specimens given 5×10^8 r. The results were compared with the results for unirradiated salt (Fig. 11).

Very little difference in the creep rate can be seen between the irradiated and unirradiated salt. Only a little over a thousandth of an inch strain separates the two curves. The unirradiated salt actually shows less strain than

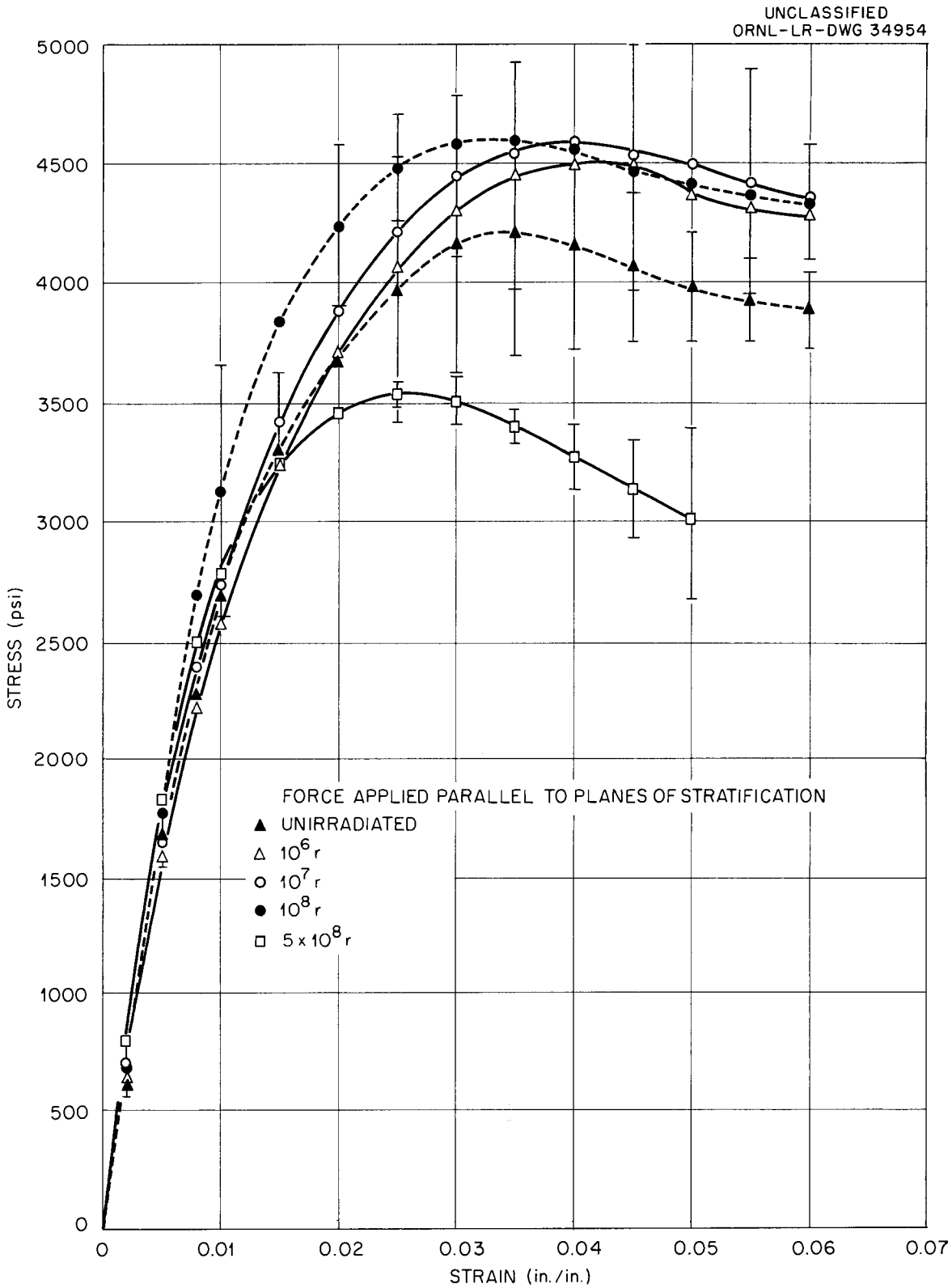

Fig. 10. Compression Test of Bedded Salt at Room Temperature.

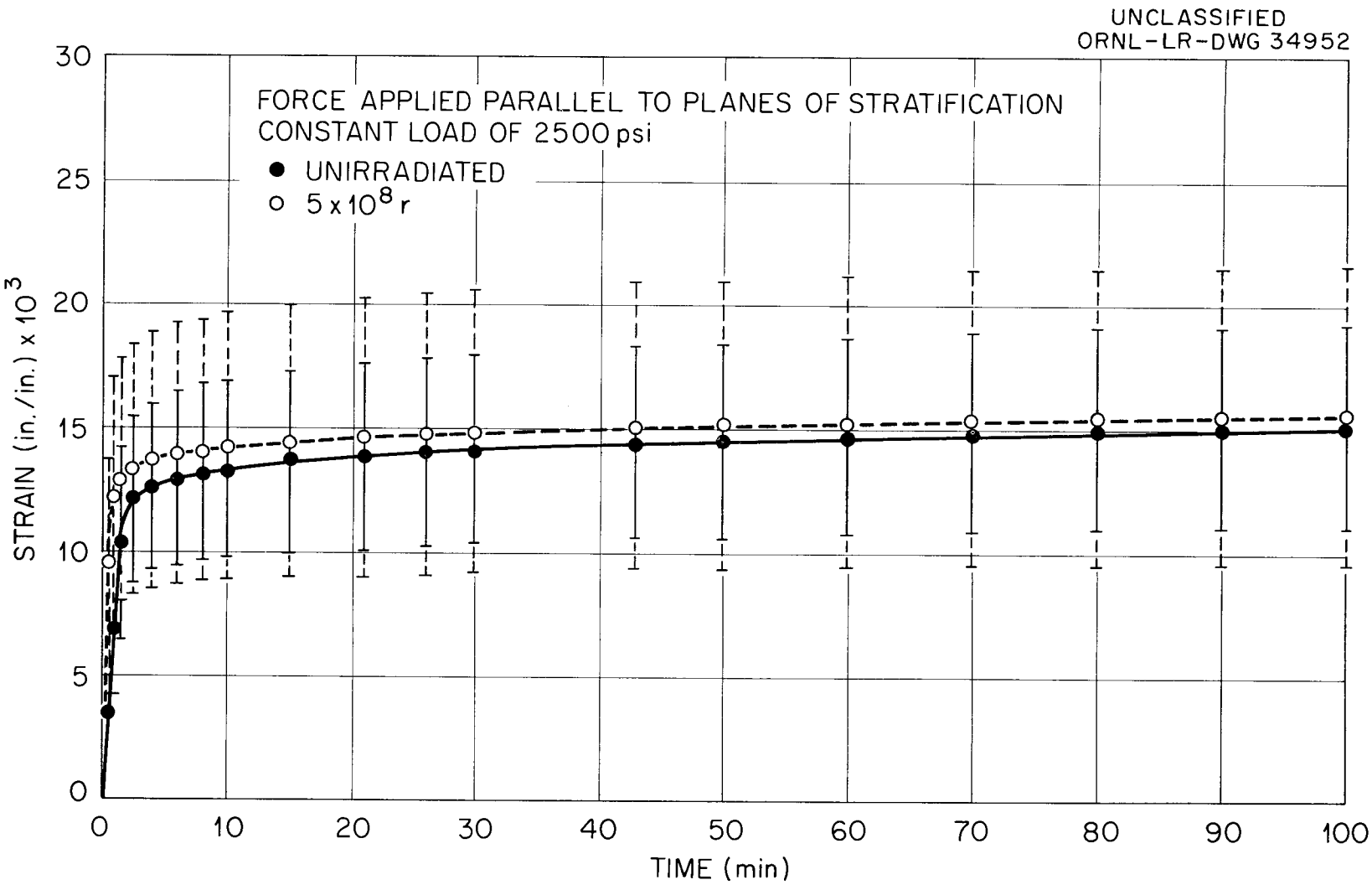

Fig. 11. Creep Test of Bedded Salt at Room Temperature.

the irradiated specimen. If the modulus of elasticity is increased, the irradiated salt should show less strain. However, two of the irradiated specimens developed cracks during the test, resulting in a wide variation in results and probably causing the greater strain.

Tests at 200° Centigrade

Stress-Strain Relationship of Irradiated Bedded Salt Loaded Parallel to the Planes of Stratification.-- The expected rise in temperature of the salt surrounding waste-filled cavities suggests the need for determining the structural properties of irradiated and nonirradiated salt at elevated temperatures. Three specimens each of bedded salt, unirradiated and exposed to 5×10^8 r, were tested at a temperature of 200° C.

The results of these tests are given in Fig. 12 and in Table 1 (note that the strain scale has been changed in Fig. 12). As expected, the plasticity increases quite rapidly with temperature, with a maximum deformation of 25%. Also, the modulus of elasticity, yield strength, and apparent elastic limit are lower than at room temperature. As in the room temperature tests, the stress-strain response at 5×10^8 r rises much faster initially and falls off more rapidly than the stress-strain response for the unirradiated specimens. This gives, as at room temperature, an increase in the modulus of elasticity, yield strength, and apparent elastic limit and a decrease in compressive strength.

Conclusions

On the basis of these tests, unirradiated dome salt is consistently stronger than unirradiated bedded salt. Under both static and creep loading conditions the unirradiated bedded salt appears to be stronger when loaded parallel to the planes of stratification than when loaded perpendicular to the

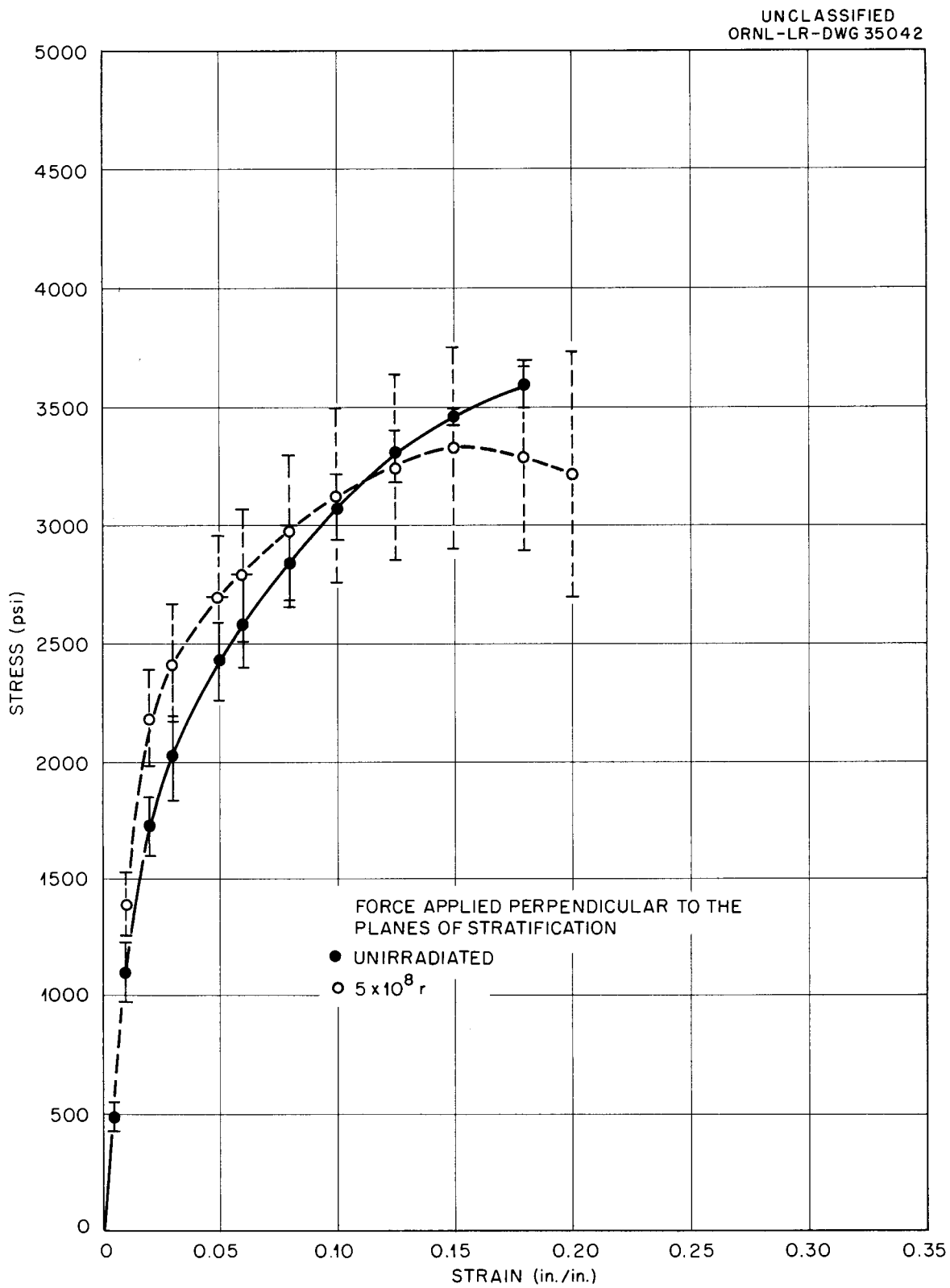

Fig. 12. Compression Tests of Bedded Salt at 200°C.

planes of stratification. However, the difference is within the statistical variation of the tests. Radiation causes only minor changes, if any, in the structural properties of salt. The most significant changes are: (1) a decrease in the compressive strength at an exposure dose of 5×10^8 r, (2) an increase in the modulus of elasticity at an exposure dose of 5×10^8 r, and (3) an increase in the yield strength and apparent elastic limit at all exposure doses except 10^6 r. However, with one exception (the compressive strength at 5×10^8 r), all are within the statistical variation of the tests. It appears that the effects of radiation on the structural properties of rock salt are minor.

CHEMICAL INTERACTION OF WASTE AND SALT

The safe disposal of radioactive wastes in rock salt is dependent in part upon the chemical interaction between the wastes and the salt. In view of the large number of variables and possible reactions in acid or neutralized waste-salt mixtures, it was felt that oxidation-reduction reactions should be investigated initially. The study of radiation-induced reactions would come later.

Unirradiated Mixtures

Nitric acid in the Purex waste and sodium chloride in the salt constitute the bulk of available reactants; however, other materials must also be considered. For example, natural salt includes associated deposits of mineral carbonates, sulfates, and other chloride salts, while the wastes contain small quantities of corrosion products and organic complexing agents.

Samples of salt were selected from the Kleer Salt Mine (Grand Saline, Texas), the International Salt Mine (Detroit, Michigan), and the Carey Salt Mine

(Hutchinson, Kansas). The Texas salt, typical of dome salt, was very pure (98%), whereas the other two samples of bedded salt ranged in purity from 87% to 94%. Synthetic, nonradioactive Purex waste solution was prepared according to ORNL CF-58-4-45.[11]

Preliminary experiments indicated that precipitation and thermal reactions between synthetic Purex waste and salt were of little importance. Gas production was found to be the most reliable index of chemical interaction. To study the effect of acidity, temperature, and salt purity on gas production, a series of acidified and neutralized waste-salt mixtures were prepared and stored in a temperature controlled ($\pm 1^{\circ}$ C) water bath. The total pressure and volume of gas produced by these mixtures at 25°, 60°, and 80° C, were measured with a gas burrette and water manometer, and collected for quantitative analysis. The results of these tests at 25° C are shown in Fig. 13. The gas produced in the Texas salt-waste mixture occurs immediately, whereas the Kansas and Michigan salt-waste mixtures produce larger volumes of gas at a much slower rate. Neutralized waste-salt mixtures produce gas at about the same rate as the control samples. These tests indicate that the system behaves according to the usual oxidation-reduction reactions, i.e., the rate of reaction increases with increases in concentration and temperature.

Quantitative and qualitative analyses of the gas showed that NO_2 was produced in trace amounts in the neutralized waste-salt mixture and in small amounts (less than 2% by volume) in the acid waste-salt mixtures. The major constituent in the gas from acid waste-salt mixtures was CO_2. If Cl_2 and NO_2Cl were produced, the concentration was below the detectable limits of the ORSAT gas analysis equipment used in these tests.

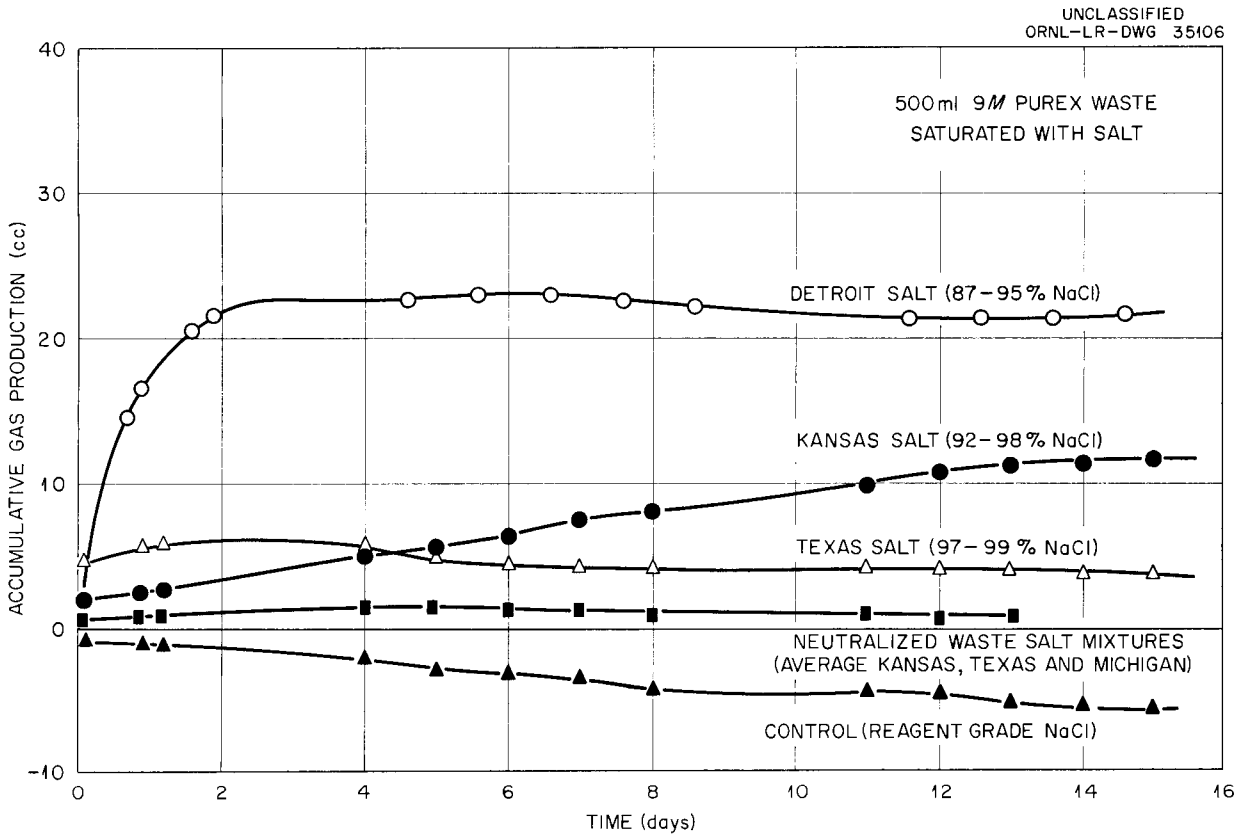

Fig. 13. Gas Production of Unirradiated Purex Type Waste.

Irradiated Mixtures

The effect of absorbed beta and gamma radiation on these reactions can best be studied by placing radioactive wastes in rock salt. However, some information can be obtained from waste-salt mixtures exposed to Co^{60} gamma radiation.

Waste-salt mixtures were placed in duplicate one-liter Pyrex bottles equipped with vent tubes. The bottles were placed in the Cobalt Storage Facility and connected to the gas measurement apparatus with 1/8-inch I. D. plastic tubing through the thermocouple wells. This arrangement permitted volume measurement and sampling during the radiation period. The recorded temperature was the average temperature of the radiation chamber. All of the gas produced during the radiation period was collected and analyzed with the ORSAT apparatus. The cumulative gas production (average of two samples) for 500-ml waste-salt mixtures exposed for 30 hr is shown in Table 2. Neutralized wastes with no salt (Run No. 4) produced the greatest volume of gas (1120 cc), while neutralized wastes saturated with salt (Runs No. 5, 8, and 10) produced the next largest volumes, 1090, 925, and 900 cc, respectively. The smallest volumes of gas, between 300 and 600 cc, were produced by the saturated acid waste (Runs 3, 7, 9, 11, and 13) and the unsaturated acid wastes (Runs 6 and 15). A cumulative record of each of these types is shown in Fig. 14. Gas production from irradiated neutralized wastes is about 90 to 100 times greater than from unirradiated wastes, and gas production from irradiated acid waste is about 15 to 30 times greater than from unirradiated waste. The difference in gas production between the saturated and unsaturated salt-waste mixtures is proportional to the differences in vapor pressure for the two mixtures (Runs 4 and 10). Qualitative analysis

Table 2. Cumulative Gas Production of Waste and Waste-Salt Mixtures Exposed to Gamma Radiation

Radiation field, 3.5×10^6 r/hr
Initial Sample Volume, 500 ml

Run	Description of Solution	Saturated with Salt	Cumulative Gas Production (cc)				
			Irradiation Time				
			5.0 hr	10.0 hr	15.0 hr	20.0 hr	30.0 hr
3A	8 M Purex, acid	Yes	143.7	220.7	328.3	420.9	562.0
4A	8 M Purex, adjusted to pH 10.0	No	275.4	443.6	622.1	811.9	1116.8
5A	8 M Purex, adjusted to pH 10.0	Yes	355.9	555.8	686.1	798.3	1086.2
6A	8 M Purex, acid	No	248.4	279.1	295.0	320.4	382.8
7A	4 M Purex, acid	Yes	221.7	230.3	234.8	248.9	
8A	4 M Purex, adjusted to pH 10.0	Yes	290.4	430.7	549.2	689.8	925.2
9A	1.88 M Purex, acid	Yes	215.6	239.9	279.1	317.1	344.2
10A	8 M Purex, adjusted to pH 10.0	Yes	264.1	420.9	547.9	651.1	902.2
11A	5 M Purex, acid	Yes	263.6	300.0	311.0	319.4	396.7
12A	6.27 M Purex, acid	Yes					
13A	8 M Purex, acid	Yes	291.6	349.8	360.6	381.0	479.0
14A	Salt slurry, 8 M Purex, acid	Yes	459.5	505.3			
15	8 M Purex, acid	No	292.8	322.1	339.3	402.7	506.0

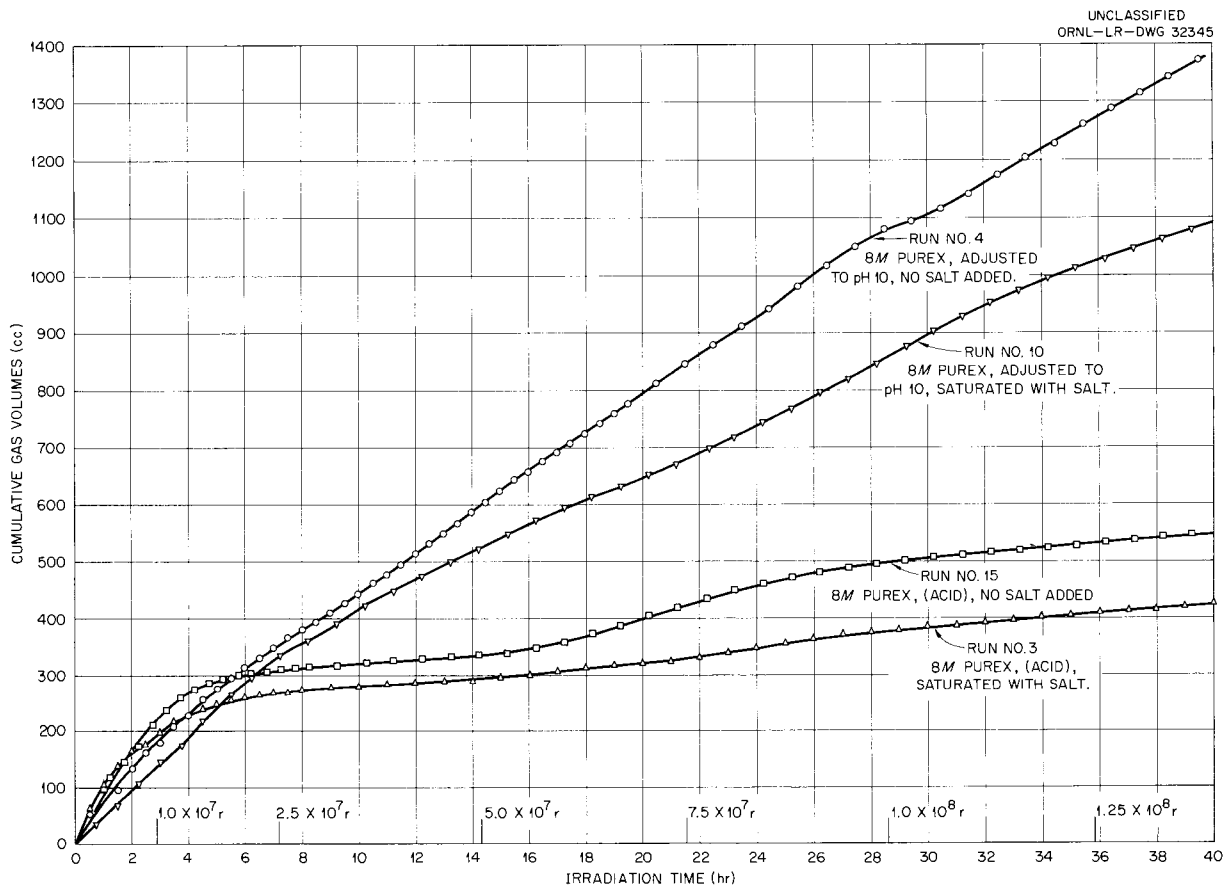

Fig. 14. Gas Production in Purex-Type Waste Subjected to Gamma Irradiation.

of the gases produced in acidic and basic solutions (Tables 3 and 4) shows very little difference in composition; however, there is an increase in hydrogen and oxygen concentrations in the neutralized solution.

These results indicate that gases produced in acidified and neutralized waste-salt mixtures are qualitatively similar and probably derived from the same reactions. The increased hydrogen and oxygen concentrations in neutralized solutions may be the result of an additional reaction or the increased radiolysis of water. One physical difference in the two mixtures, which seems to correlate with increased gas production, is the presence of a hydrous iron oxide precipitate in the neutralized solutions. This precipitate increases the concentration of solid reactants available for gamma ray interaction which may increase the number of secondary electrons available for chemical interaction.

Model of the Field Test

Attempts were made to use small samples of salt (1- and 2-inch cubes) as experimental storage containers for acid and neutralized waste solutions. Invariably, however, these small samples of mined salt were found to have cracks or fractures and could not be used. Larger salt blocks (~ 2 ft on a side), as shown in Fig. 15, with hand-excavated cavities approximately 7 1/2 inches square and 9 1/2 inches deep were used instead. A Teflon cover, reflux condenser, 200-watt quartz-sheathed immersion heater, and thermocouples located in the cavity, as well as in the salt block, (locations given in Table 5) were used to test a small-scale version of a field experiment. Six liters of 7 molar, neutralized, synthetic Purex waste, saturated with the salt removed from the cavity, were poured into the cavity. The temperature of the liquor was raised

Table 3. Chemical Composition of Gas Produced During Run 15

8 M Purex, Acid, No Salt Added

Radiation field, 3.5×10^6 r/hr

Irradiation Time (hr)	Sample Volume (cc)	Composition (%)				
		Cl_2	NO_2	O_2	H_2	N_2
0.5	59.5	1.2	1.6	21.6	0.3	75.2
2.25	98.7	0.9	0.2	20.6	0.0	78.3
4.75	97.0	0.4	0.2	20.9	0.1	78.4
17.25	96.2	0.4	0.2	21.8	2.9	74.6
22.25	67.8	0.4	0.9	24.9	1.2	72.6
28.25	81.9	1.8	1.7	26.3	2.3	67.9
41.25	56.5	1.2	1.4	25.0	2.3	70.2
48.60	43.3	1.6	1.0	25.5	3.0	69.1

Table 4. Chemical Composition of Gas Produced During Run 10B

8 M Purex, adjusted to pH 10, saturated with salt

Radiation field, 3.5×10^6 r/hr

Irradiation Time (hr)	Sample Volume (cc)	Composition (%)				
		Cl_2	NO_2	O_2	H_2	N_2
0.83	60.3	0	0	19.6	0	80.4
3.83	97.0	0	0	21.1	0	78.9
5.58	99.0	0	0	28.2	0	71.8
8.33	95.8	0.5	0	28.9	0.6	69.9
11.33	94.3	0	0	34.0	0.5	65.4
16.33	94.8	0	0	41.0	1.2	57.9
20.33	80.3	Trace	0.5	41.5	1.6	56.3
26.33	39.8	0	0.2	53.0	2.3	44.5
29.33	68.4	0.3	3.8	57.2	3.6	35.1
35.33	98.2	Trace	1.1	70.1	3.6	25.0
43.33	94.8	0	7.5	59.4	3.9	29.3
46.58	58.9	0	13.7	41.5	1.4	41.5
53.33	88.7	0	3.2	70.1	4.5	22.2
66.33	97.6	0	5.0	70.7	4.0	20.3
70.75	96.2	0	1.0	44.1	1.9	52.6

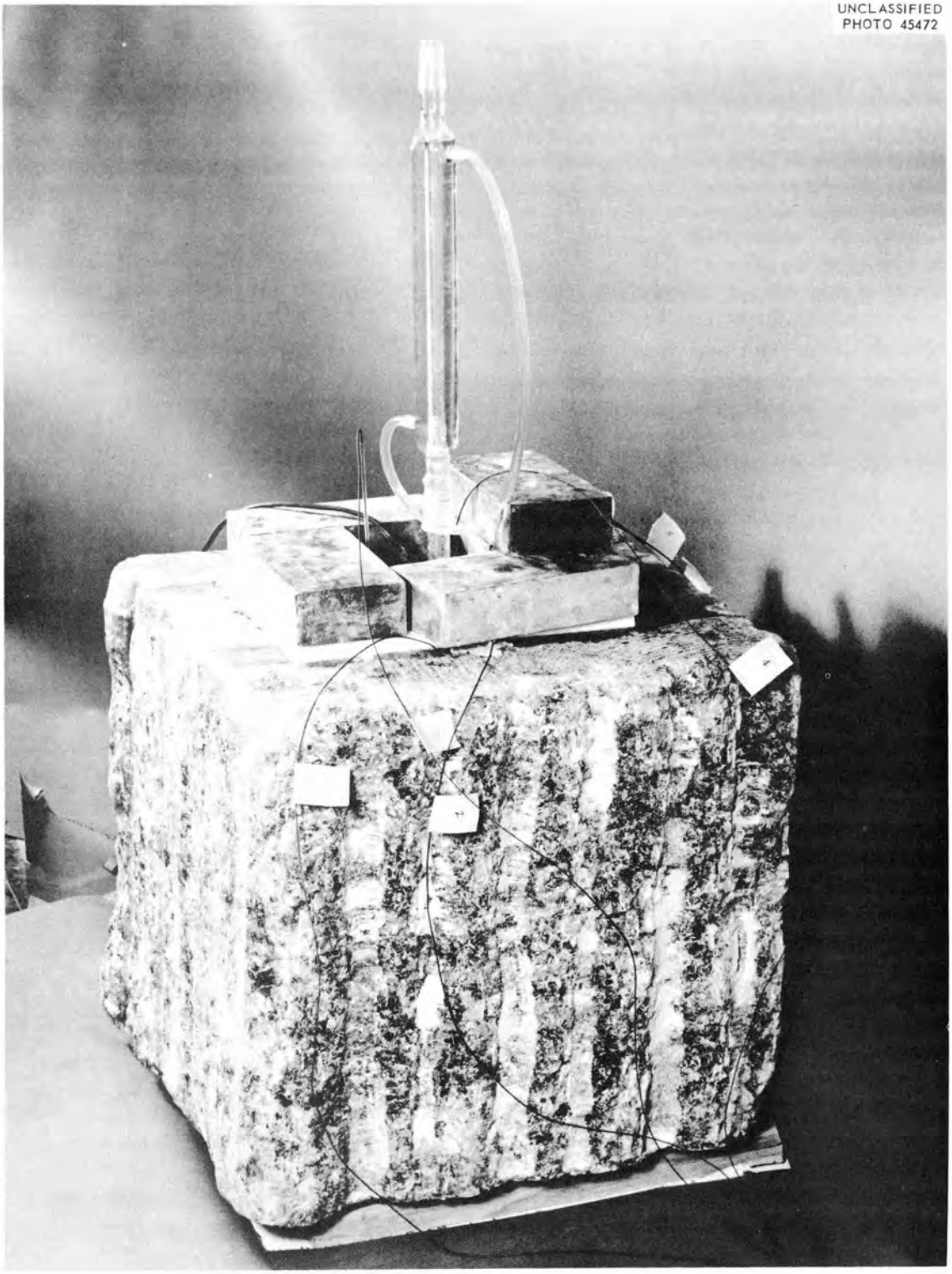

Fig. 15. Model of Field Experiment.

Table 5. Thermocouple Locations

TC	X*	Y	Z
1	16	3	7 1/2
2	16	10	10
3	10	5	8
4	10	3	11
5	4	5	11 1/2
6	4	5	8
7	10	12	11
Block	0 - 20	0 - 20	0 - 20
Cavity	6.25 - 13.75	0 - 9.5	6.25 - 13.75
Heater	10	4 - 7	10

* X, Y, and Z equal zero at top left front corner of block.

to 90° C. The recorded temperatures of thermocouples 3 and 4 (Fig. 16) show a small thermal gradient in the liquor. Evidently the convective currents are not sufficient to equalize the temperatures throughout the liquor. The maximum temperature recorded out in the block was 50° C. Temperatures in various parts of the block show that there is no difference in the thermal conductivity-heat flux ratio in any part of the block.

During the course of the experiment recrystallized salt appeared in No. 2 and No. 5 thermocouple holes at the outside surface of the block. The recrystallized salt also appeared at various other locations on the surface. Salt continued to crystallize, and the growths increased during the course of the experiment. The growths occurred both above and below the liquid level in the cavity. After two months the block was permitted to cool, the cavity equipment and wastes were removed, and then the block was broken apart for examination. Five liters of the original six liters of waste solution were recovered. The upper 2 1/2 inches of the cavity was enlarged significantly from its original dimensions (Fig. 17).

A thin layer of hydrous iron oxide (< 1/16 inch thick) from the neutralized wastes coated the lower 7 inches of the cavity walls below the liquid level. Apparently, the wastes did not penetrate this coating. A layer of recrystallized salt about 2 inches thick "bridged" the cavity above the liquid level. Salt, in the upper 2 1/2 inches of the cavity immediately below the cover, was dissolved by the condensing water vapor and was recrystallized as the brine moved down the exposed side walls of the cavity to form a firm bed of crystals over the liquor.

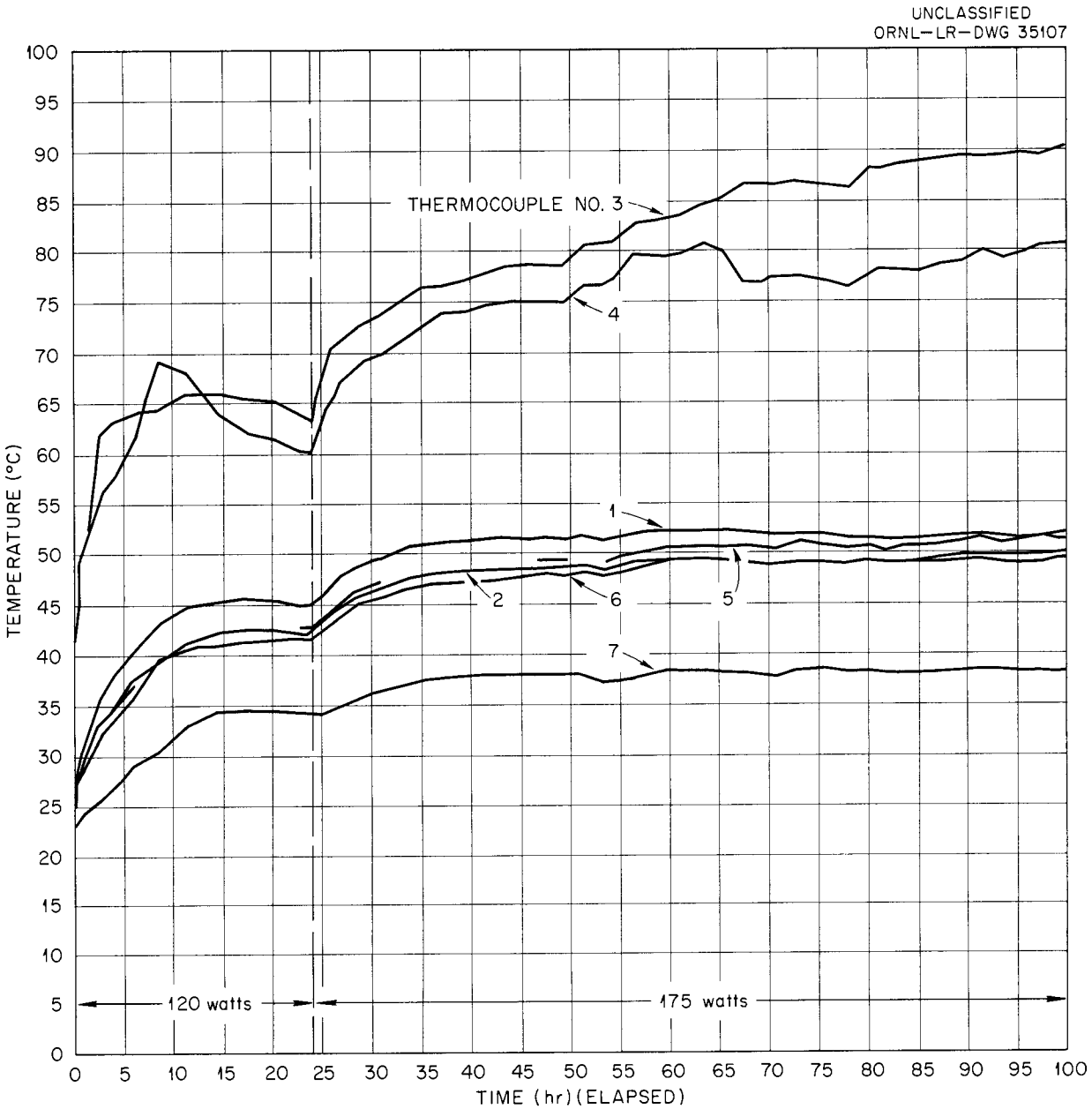

Fig. 16. Temperature Distribution in Salt Block.

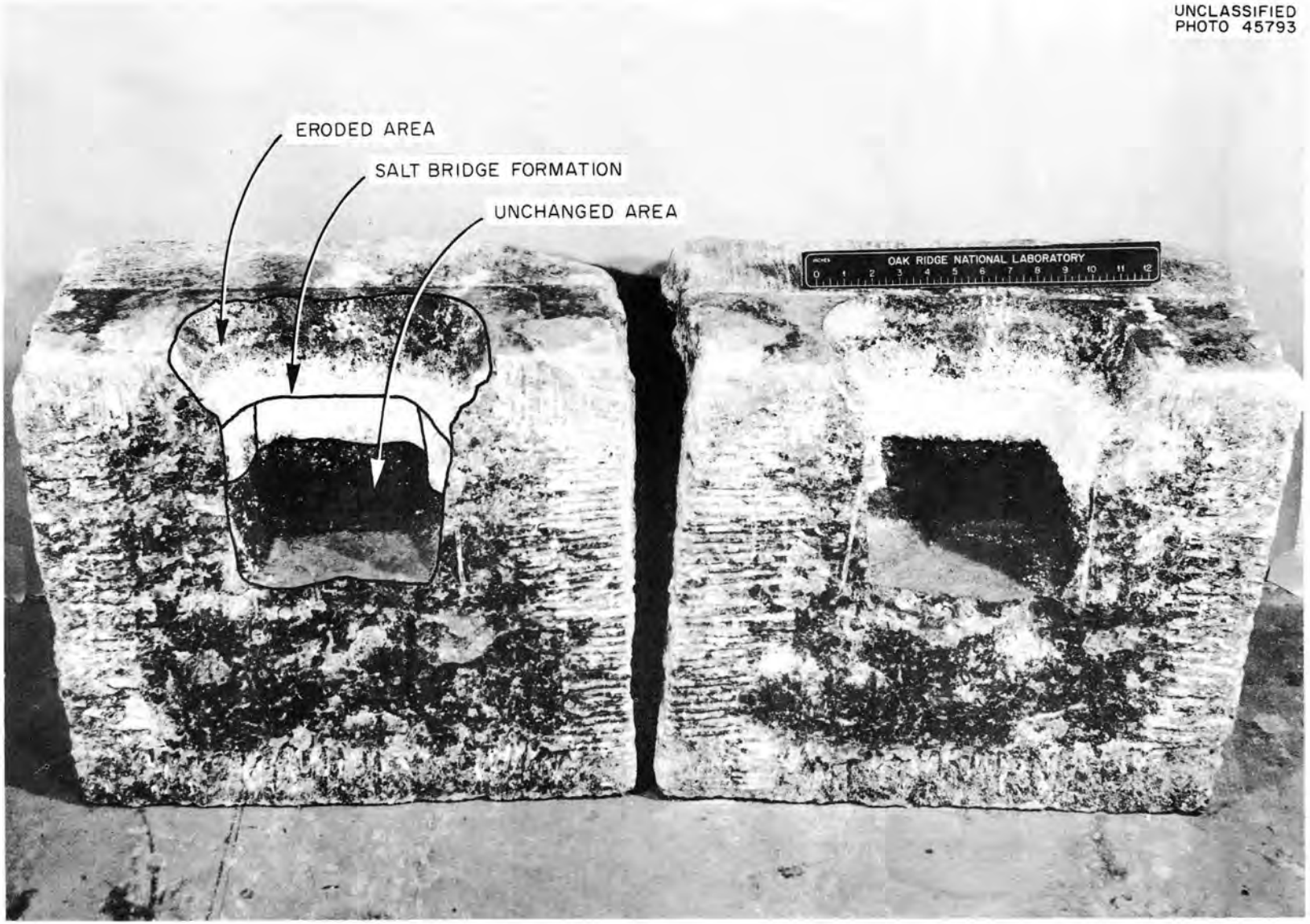

Fig. 17. Results of Tests on Model of Field Experiment.

FIELD TESTS

In the laboratory, it is practically impossible to reproduce the conditions that exist in a natural rock salt formation. Samples removed from the formation go through a process of stress relief, violent enough in some cases to shatter the sample. Even if the samples are not shattered, there is some alteration of the physical properties of the rock. If the overburden pressure is reapplied, the stresses induced will not necessarily be the same unless the sample is completely elastic, which is not true for geologic materials. Therefore, it is necessary to conduct field experiments before a prototype disposal facility can be designed.

Experimental Design

Information can be obtained in the field experiment regarding (1) the structural integrity of the cavity; (2) the migration of nuclides from the cavity; (3) the migration of the cavity itself, due to the condensation of the water vapor on the surfaces causing the salt to go into solution and to flow back into the waste cavity; (4) temperature gradients, both inside and outside the cavity; and (5) gas production.

Eight tests are planned requiring eleven excavations. Each test will run for approximately six months. Since some tests will run concurrently, the over-all field test program will require about two years. Major variables to be studied include: (1) the type of waste (Purex and acid aluminum nitrate), (2) the concentration and pH of the wastes, (3) the geometrical configuration of the cavity, (4) the surface area/volume relationships, (5) the method of construction (hand excavated, blasted, or solution-mined), and (6) the rate of

heat production.

Site Selection

The first tests with synthetic wastes and simulated fission product decay heating will be followed by tests with actual radioactive wastes. These preliminary nonradioactive tests will be conducted in mined-out space in some salt mine.

During the past year, a Laboratory subcontractor (Geotechnical Corporation, Dallas) investigated operating salt mines in the northeastern section of the country. The investigation was limited to this area, because bedded deposits are found in this area, and it seemed likely to be the general location of the first commercial fuel-reprocessing plant. It was found that the owners were amenable to the use of their mines for the field tests, since the first tests would not involve the use of radioactive wastes. There would be a number of advantages in using mined-out space for these tests, e.g., the immediate availability of a test site, a competent work force with the necessary equipment available to excavate the cavities, and the necessary auxiliaries - shaft, hoist, electrical power, railroad sidings, etc.

The Carey Salt Company (Hutchinson, Kansas) and International Salt Companies (Detroit, Michigan) expressed their willingness to participate in these tests. Their mines satisfied most of the requirements of a good test site. A detailed analysis of the geology, geographic location, facilities in the mine, estimated costs, and the possible location of future tests indicated that the Hutchinson mine would be more desirable.

Accordingly, the Laboratory entered into contractural arrangements with the Carey Salt Company for the use of an unoccupied section of their mine to

carry out these experiments. The first tests will begin in July, 1959.

Engineering Design

The proposed test facility is shown in Fig. 18. The cavity is 7 1/2 ft square and 10 ft deep, a size that will give a surface area equal to the surface area of a 10-foot diameter sphere, when the cavity is filled to the 7 1/2-foot level. Thus, if thermal data from the field experiment agree with theoretical heat calculations, the scaling factors can be computed. Jakob has shown that the thermal distribution for small spheres and small cubes is the same.[12] The facility includes the cavity, immersion heaters, a gas tight cover, and measuring devices and recorders. All data, with the exception of liquid level, will be on strip-chart recorders. An alarm system set to function on high temperature, moisture penetration, high pressure, and loss of electrode power will be installed in the experimental area and telemetered to the mine office.

All equipment will be prefabricated to sizes that can be brought down the shaft and assembled in the mine. The cavity will be excavated in the floor of an existing room in the mine. Conventional room and pillar mining has been used in the Hutchinson mine, the pillars being 50 ft by 50 ft and the rooms, 50 ft by 50 ft. The floor-to-ceiling height varies from 10 to 12 ft. In older sections of the mine, the rooms are 50 ft by 300 ft and with 6-foot-high pillars, 20 ft by 300 ft. Core drilling in the proposed test area of the mine has shown that the salt bed beneath the present floor exceeds 30 ft in thickness.

The first two cavities will be hand-excavated to avoid the shattered zones caused by blasting. Blasting operations appear to disturb and shatter the rock to a depth of 12 to 18 inches.

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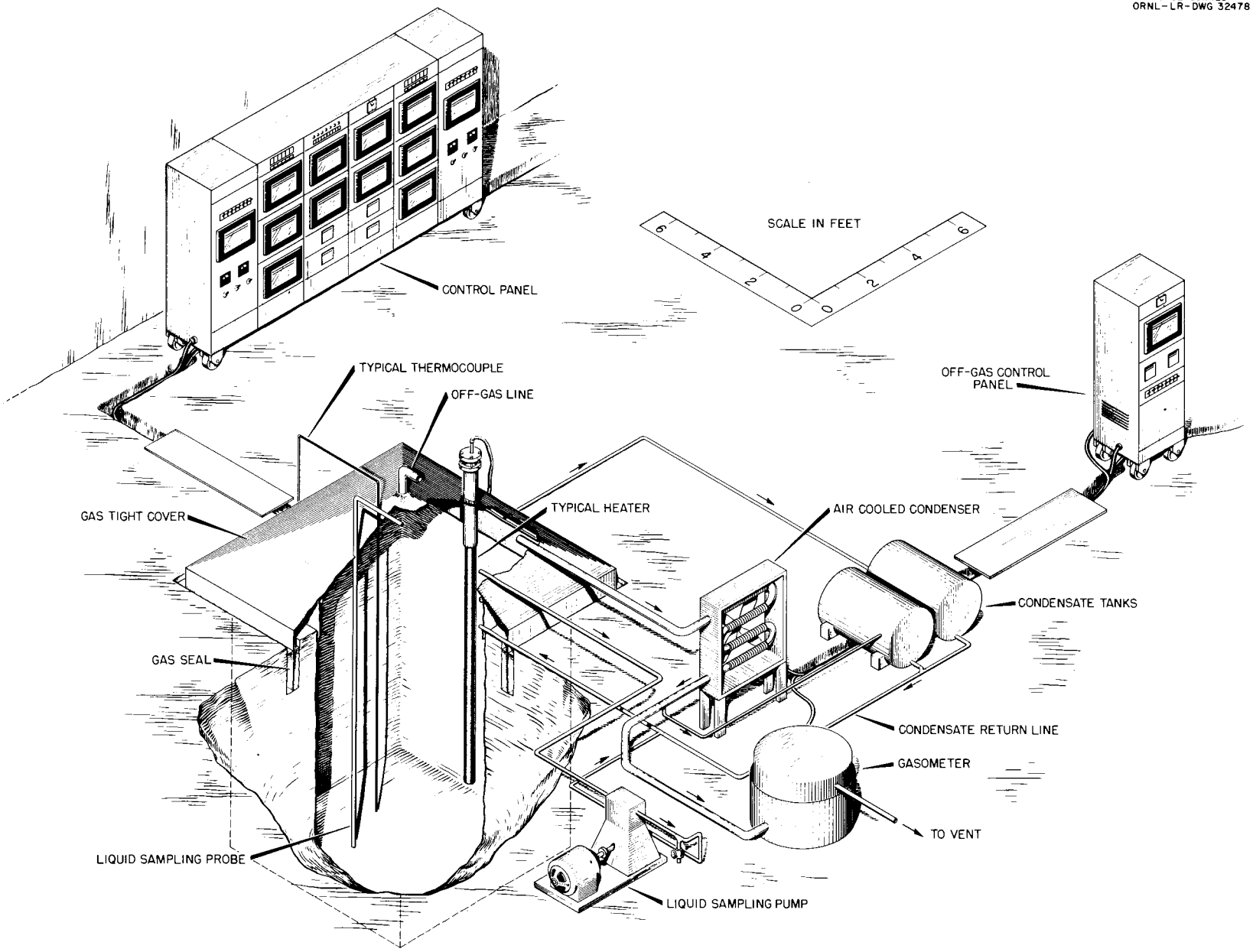

Fig. 18. Proposed Salt Field Experiment.

Conductivity probes will be placed next to the cavity to measure any moisture changes in the salt. The electrical conductivity of salt is extremely sensitive to changes in moisture content; so the changes in electrical conductivity will indicate any movement of the wastes out into the surrounding salt. A gas tight cover will be installed so that off-gases can be collected, condensed, measured, analyzed, and returned to the cavity. As discussed previously, the total quantity of gas produced will be small.

Heat must be provided to simulate the heat of radioactive decay. It is, of course, impossible to duplicate exactly the heat generated by fission-product decay where each nuclide produces heat as a function of its energy and half life. However, the calculated temperature rise is based on the assumption that the waste is uniformly mixed, so uniform heat production is desired. In the case of neutralized wastes, more fission products are in the precipitate than in solution, so additional heat must be distributed in the precipitate. Thermocouples will be placed in the cavity (in both liquid and gaseous phases) and in the salt surrounding the cavity.

Operating Procedure

After completion of construction and pretesting, about 7,000 gal of synthetic waste (saturated with salt) will be transferred to the two test cavities. When the waste solution temperature has reached the ambient mine temperature, the heaters will be turned "ON," and the power varied to correspond to a predetermined fission product decay function (representative of a given waste type) while considering the decreasing resistivity of the solution with increasing temperature.

Temperatures in the cavity (above and below the liquid level) and in the salt surrounding the cavity will be recorded continuously. Also, heater power and electrical conductivity of the surrounding salt will be recorded continuously. Periodic samples of cavity solution, condensate and off-gases will be withdrawn and analyzed. Charts from the recorders will be removed and replaced as needed and routine preventative maintenance of the recorders and power controls will be performed at regular intervals.

The peak temperature of the cavity solution ($\sim 80^{\circ}$ C) is expected to occur in approximately five months. Once the solution temperature levels off, the heaters will be turned "OFF," and the solution will be cooled to ambient temperature. After removal of the cavity cover and heaters, the wastes will be disposed of in a nearby solution cavity. The empty cavities will then be filled with rock salt and the equipment moved to the next test cavities.

MATERIALS STUDY

A critical design problem has been that of finding suitable materials for the fabrication of heaters, cavity covers, and off-gas equipment because of the corrosiveness of waste-salt mixtures and the associated off-gases. In the actual storage of radioactive wastes, this will not be a serious problem.

Corrosion Studies

The wastes themselves are not highly corrosive. Both acid aluminum nitrate and Purex (nitric acid) wastes are readily handled in austenitic stainless steels (300 series) or, when neutralized with sodium hydroxide, in ordinary steel. However, when the acid solutions are saturated with common salt (NaCl),

the resulting solutions and vapors are extremely corrosive. The high corrosion rates in the nitric acid-sodium chloride solution are due to the presence of the same ions as are present in a nitric acid-hydrochloric acid (aqua regia) solution. Due to the presence of nascent chlorine, aqua regia is one of the most corrosive materials known, readily dissolving both platinum and gold, as well as normal metallic materials. Corrosion rates significantly greater than 0.001 inches/year are considered to be unsatisfactory.

Table 6 presents the results of a study of possible construction materials. The waste of immediate interest is Purex waste under acid conditions, since this is the most abundant present-day waste. The table shows that only a limited number of materials are suitable. Titanium is considered the most resistant material on the basis of corrosion tests carried out at Battelle Memorial Institute in connection with Darex process development.[13] These were tests in HNO_3 acid containing HCl, or with HCl gas bubbling through the liquid. A second metal which shows promise is a cobalt base alloy, Haynes 25 (Haynes Stellite Co.). The corrosion rate was negligible in 77 days at 80° C with the sample suspended at the interface of the waste and vapor under reflux conditions. There was no evidence of pitting. Other samples containing welds showed higher corrosion rates.

The nonmetallic, inorganic materials considered include graphite and glass-lined steel. Graphite is brittle and involves some limitations in fabrication, since it cannot be welded. A glass lining applied to a cover fabricated of steel would be satisfactory under acid conditions. However, glass and other siliceous materials are appreciably attacked by strong NaOH present in the neutralized waste.

Mong organic materials, Teflon (E. I. Du Pont de Nemours and Co., Inc.) appears to be completely resistant. The cost of the material, combined with

Table 6. Corrosion Data in Salt Saturated Simulated Waste

Material	Corrosive Medium (salt saturated)	Temperature °C	Exposure Days	Corrosion Rate (in./yr)	Remarks
410 SSt	Hope	Approx. 22°	238	----	Failed.
410 SSt	Neut. Hope	Approx. 22°	238	Negl.	
410 SSt	Purex-7MHNO$_3$	Approx. 22°	238	----	Failed.
410 SSt	Purex-Neut.	Approx. 22°	238	Negl.	
316 SSt	Hope	Approx. 22°	238	Negl.	
316 SSt	Hope-Neut.	Approx. 22°	238	Negl.	
316 SSt	Purex-7MHNO$_3$	Approx. 22°	238	----	Failed.
316 SSt	Purex-Neut.	Approx. 22°	238	Negl.	
347 SSt	Hope	Approx. 22°	238	0.0025	
347 SSt	Hope-Neut.	Approx. 22°	238	Negl.	
347 SSt	Purex-7MHNO$_3$	Approx. 22°	238	----	Failed.
347 SSt	Purex-Neut.	Approx. 22°	238	Negl.	
Inconel	Hope	Approx. 22°	238	0.013	
Inconel	Hope-Neut.	Approx. 22°	238	Negl.	
Inconel	Purex-7MHNO$_3$	Approx. 22°	238	----	Failed.
Inconel	Purex-Neut.	Approx. 22°	238	Negl.	
Inor	Hope	Approx. 22°	238	0.008	
Inor	Hope-Neut.	Approx. 22°	238	Negl.	
Inor	Purex-7MHNO$_3$	Approx. 22°	238	----	Failed.
Inor	Purex-Neut.	Approx. 22°	238	Negl.	
Hastelloy C	Purex-7MHNO$_3$	Approx. 22°	139	0.0004	
Hastelloy C	Purex-7MHNO$_3$	Approx. 80°	19	----	Failed.
Haynes 25	Purex-7MHNO$_3$	Approx. 80°	77	0.0007	Stamped sample.
Haynes 25	Purex-7MHNO$_3$	Approx. 80°	34	0.00147	Sample cut from sch. 40 pipe.
Haynes 25	Purex-7MHNO$_3$	Approx. 80°	34	0.004	Sample from sch. 40 pipe containing butt weld.
Haynes 25	Purex-Neut.	Approx. 80°	77	Negl.	Stamped sample.
Titanium	Purex-7MHNO$_3$	Approx. 80°	63	0.0008	
Titanium	Purex-Neut.	Approx. 80°	63	Negl.	
Titanium	Purex-7MHNO$_3$	Approx. 22°	127	Negl.	
Teflon	Purex-7MHNO$_3$	Approx. 100°	70	----	No change. Slight weight gain.

Table 6. Corrosion Data in Salt Saturated Simulated Waste (continued)

Material	Corrosive Medium (salt saturated)	Temperature °C	Exposure Days	Corrosion Rate (in./yr)	Remarks
Polyvinyl Chloride (unplasticized)	Vapor above Purex-7MHNO$_3$	Approx. 80°	24	Negl.	Approx. 1.4% weight gain without apparent swelling.
Polyvinyl Chloride	Purex-7MHNO$_3$	Approx. 80°	21	Negl.	Totally immersed in medium. One per cent gain without apparent swelling.
Graphite	Purex-Neut.	Approx. 80°	30	----	No corrosion. Slight weight gain.
Graphite	Purex-7MHNO$_3$	Approx. 80°	30	----	No change. No weight loss during use of graphite as electrode in heating experiments.

1. All samples exposed partially immersed, except as noted.
2. Samples at 80° C (and higher) under refluxing conditions.

the problems of fabricating a large cover assembly (approximately 10 ft by 10 ft by 3 ft high) makes it unattractive. The possibility of bonding thin Teflon to a steel cover is being studied. Unplasticized polyvinyl chloride may be suitable for some of the experimental equipment though it exhibits some swelling in the acid liquid.

Heater System

Uniform heat production in the solution is desirable in the field cavities. It may be achieved by covering opposite walls of the cavity with flat plate electrodes and applying a voltage between them. The resulting current flow through the solution (electrolytic heating) will thus produce uniform heating. If the two plates are perforated and moved away from the walls slightly, the solution will have free access to the walls, producing a uniform temperature throughout.

Tests indicate that conventional immersion heaters in the acid waste would not withstand the corrosive conditions. Corrosion is not a serious problem in neutralized waste, but because of viscosity of the neutralized precipitate, thermal convection currents cannot be relied upon to equalize the heat distribution. Laboratory experiments indicate that a reasonably uniform heat distribution in the precipitate cannot be obtained readily with immersion heaters. Therefore, the wastes will be heated electrolytically.

Electrolytic heating introduces problems of (1) the design of the electrodes, (2) the design of the power supply, and (3) possible gassing due to electrolysis.

Electrodes.-- Almost any corrosion resistant material should be suitable for use as electrodes in the neutralized waste solution, since neutralized

waste is not very corrosive. However, the choice of materials for use in the acid waste is limited. Carbon, or graphite, seem to be the most desirable from the standpoint of corrosion resistance, as well as cost and availability. However, graphite is rather brittle and would be fragile in a perforated plate of the proper dimensions.

Since the salt-saturated acid waste has a fairly low viscosity it would be expected that thermal convection currents would tend to produce a uniform temperature throughout the solution, even with nonuniform heating. Several arrangements of cylindrical graphite electrodes were tried on a scale of one inch to the foot in an attempt to produce uniform heating. The electrode configuration adopted produced the power (rate of heat production) distribution shown in Fig. 19. When the salt solution temperature was raised 10° C by this electrode arrangement the temperature variation to within one-half inch of the walls and the electrodes was only one degree, indicating a relatively uniform heat distribution.

Power Supply.-- The electrical resistivities (reciprocal of conductivity) of the salt-saturated acid waste, the neutralized precipitate, and the neutralized supernate measured by an Industrial Instruments Company Conductivity Bridge are shown in Table 7. It may be observed that the neutralized waste has a resistivity at 20° C, nearly three times that of the acid waste, but at 80° C the ratio drops to only a little greater than two. Thus, the voltage and current range of the power supply required to heat the neutralized waste will be greater than that required by the acid waste.

The electrical resistivities of both the acid and the neutralized wastes are relatively low and, to produce the desired power in the solution, will

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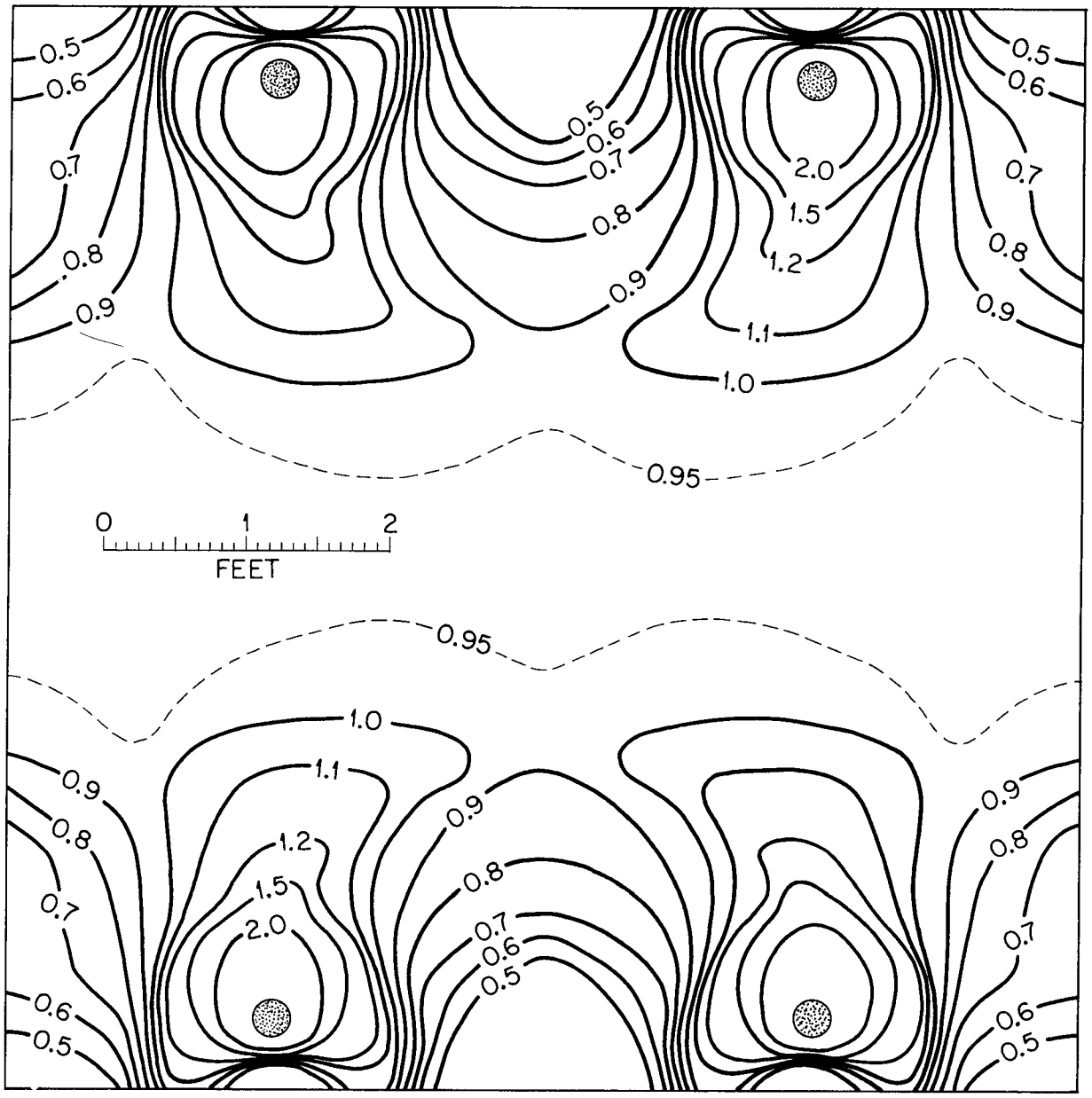

CONSTANT POWER PRODUCTION CONTOURS
ARBITRARY POWER SCALE

Fig. 19. Power Distribution with Four Electrodes.

Table 7. Waste Solution Resistivity

Solution	Resistivity (ohm-cm)	
	20° C	80° C
Salt Saturated Acid	2.4	1.2
Salt Saturated Neutralized Precipitate	6.8	2.6
Salt Saturated Neutralized Supernate	6.2	2.4

require a high-current, low-voltage power supply. Since the resistivities of the solution drop rapidly with increasing temperature, decreasing voltage and increasing current will be necessary in order to maintain a given power input. Fission product power production decreases with time and will tend to reduce the increased current demand caused by increasing temperature, but will call for a further decrease in voltage.

The initial voltages required for the two types of waste, however, are not directly indicated by the ratio of their 20° C resistivities, due to two factors. One is that the use of carbon rod electrodes in the cavity filled with acid waste will produce a total effective solution resistance which is greater than that which would be obtained with plate electrodes, thus requiring a greater voltage to produce the same power. The other factor is that from 70 to 90% of the fission product radioactivity in the neutralized waste will be in the precipitate, thus requiring most of the power in the precipitate. To meet this requirement, the plate electrodes will be in the precipitate only and not extend up into the supernate. This results in a greater effective resistance and consequent increase in required electrode voltage.

Tests using carbon electrodes in approximately two and one-half gallons of salt-saturated acid waste at several temperatures and power inputs showed that the wattage input was essentially the same as the volt-ampere product, and thus there is no reactive current capacity which must be built into the supply.

The Hutchinson mine has 2300 volt, 60 cycle, 3 phase power available. This will be stepped down in voltage, and each experiment will be connected to a separate phase, since the electrodes need to run on single phase power to prevent sacrifice of the graphite.[14] One way to obtain the necessary flexibility is to step down to 440 volts before feeding into a Powerstat or other

variable transformer and then, with a special transformer, step down further to the voltage necessary at the electrodes.

The approximate voltage and current ranges for the acid waste experiment will be from 25 volts and 800 amperes at 20° C to 10 volts and 2000 amperes at 80° C. For the neutralized waste the approximate range will be 40 volts and 500 amperes to 15 volts and 1350 amperes.

Further experiments are planned in order to obtain accurate figures for scale up calculations on the power supply before equipment is obtained.

Gassing.-- The passage of an alternating current through a conducting solution will produce gas at the electrodes if the current density at the electrodes is great enough. Tests of carbon electrodes at current densities greater than those that will be encountered in the field experiment have shown no visible gassing.

Further tests will be run to establish the actual gas production rate, if any, so that a correction may be made in the off-gas analysis of the field experiments.

CONCLUSION

This status report has described analytical work on the thermal problem associated with disposal of radioactive wastes in salt, the experimental studies of the effect of radiation on the structural properties of rock salt, and the chemical interactions between wastes and salt. The selection of a test site, the design of the field test, and the material studies associated with the field tests are also described.

The results of the work have been applied to the design of a series of tests leading to the final test, actual disposal of radioactive waste in a salt cavity. Theoretical calculations and laboratory scale experiments with disturbed samples can only give us an approximation of the results to be expected in the field. Similarly, the preliminary field tests can only simulate heat generation and chemical compatibility. The definitive test will be the actual disposal of radioactive wastes under carefully controlled conditions in some remote and isolated salt cavity.

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