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CHEMICAL APPLICATIONS OF NUCLEAR EXPLOSIONS (CANE):

PROGRESS REPORT FOR APRIL 1 TO JUNE 30, 1967

Compiled by:

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ABSTRACT

During the dissolution of ^{106}Ru -contaminated cement copper in sulfuric acid, about $2/3$ of the ruthenium dissolved in the acid solution and the other $1/3$ remained in the insoluble sludge. Only about 0.3% of the soluble ruthenium deposited with 13% of the copper in the electrowinning of copper from this solution. More than 80% of the ruthenium was removed from the electrolyte solution by oxidation at 80°C with sodium persulfate.

After shale oil had been heated with debris from a recent underground test shot, it was found to be contaminated with radionuclides, principally $^{95}\text{Zr-Nb}$ and ^{103}Ru . The contamination of the oil increased with increased heating temperatures.

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1. INTRODUCTION

The purpose of this program is to conduct research, development, and evaluation studies as needed in selected areas of the Plowshare Program, especially those areas concerned principally with chemistry and chemical or metallurgical engineering. The emphasis placed on the various areas of study at ORNL is related directly to the planning and scheduling of the overall Plowshare Program.

Projects which are receiving attention at this time and which are described in this report include (1) studies of the distribution and possible fate of radionuclides formed during the use of nuclear devices to aid in the recovery of copper from ore deposits, (2) studies of the distribution and possible fate of radionuclides formed during the use of nuclear devices to aid in the recovery of oil from oil shales and (3) thermal analysis of americium compounds.

Another project that is currently receiving attention at ORNL is the study of hypervelocity jet sampling as a means of removing a specimen after irradiation in the neutron flux of a detonation. A burner is being tested which may be suitable for reducing the volume of the wood that was used to catch the jetted targets in the final experiments at Vincentown, N. J.

2. NUCLEAR EXPLOSIVES IN COPPER ORE PROCESSING

D. J. Crouse, W. D. Arnold, W. B. Howerton

Fracturing of copper ore deposits with nuclear explosives, followed by in-situ leaching is being studied for the AEC by Lawrence Radiation Laboratory (LRL) and by the U. S. Bureau of Mines at Tucson, Arizona. The Oak Ridge National Laboratory is cooperating in this program by studying potential problems that might arise from the introduction of radioactive contaminants into the processing cycle. The proposed flowsheet for recovering copper involves percolating dilute sulfuric acid down through the nuclear-broken ore to dissolve the copper, collection of leach liquor at the bottom of the ore body and pumping it to the surface, recovery of a copper concentrate from the solution by cementation on iron, and recycling of the barren solution to leaching after fortifying it with acid. The test results reported here were obtained

in studies of 1) the purification of cement copper by dissolution and electrolysis, and 2) the removal of ruthenium from electrolyte solutions by oxidation with sodium persulfate.

2.1 Purification of Cement Copper

Cement copper can be purified by dissolving it in dilute sulfuric acid and recovering copper metal from the solution by electrowinning. This is an alternative to the usual method in which the cement copper is smelted into a consumable anode and then converted electrolytically to pure copper.

When ^{106}Ru -contaminated cement copper was dissolved in 1 N H_2SO_4 , about 2/3 of the ruthenium dissolved in the acid solution (Table 1). About 0.5% of the cement copper was insoluble in the acid solution and settled out as a finely-divided black sludge that contained the other 1/3 of the ruthenium. In this test, 2.5 g of cement copper (prepared by cementation from a ^{106}Ru -traced synthetic copper leach liquor) was heated for 24 hr at 80°C with 100 ml of 1 M H_2SO_4 through which pre-heated air was bubbled. The air passed through a reflux condenser and two NaOH scrubbers before leaving the system. None of the ruthenium was found to be volatilized. The final solution contained 25 g Cu per liter, and 1.2×10^4 counts $\text{min}^{-1} \text{ ml}^{-1}$ of ^{106}Ru . When this solution was electrolyzed for 6 hr at a current density of about 10 amp/ft² and a potential of about 2 volts, about 12.7% of the copper and 0.32% of the ruthenium were deposited on the cathode. Both of the electrodes were platinum and the cell temperature was 55°C. These results are similar to those reported earlier¹ for the smelting and electrolytic purification of cement copper. In those tests, there was no separation of ruthenium and copper in the smelting operation. However, in the electrolytic purification of the anode, about 2/3 of the ruthenium released from the anode dissolved in the electrolyte and about 1/3 of it was in the anode "mud." The copper product contained 75% of the copper and 1 to 2% of the ruthenium initially present in the anode. Starting with the same cement copper, either of these purification methods would apparently yield a cathode copper product contaminated with about the same amount of ^{106}Ru .

Table 1. Ruthenium Behavior in Dissolution of Cement Copper and Electrowinning

Procedure: 2.5 g cement copper (contaminated with 6.85×10^5 counts min^{-1} of ^{106}Ru per gram) dissolved in 100 ml 1 N H_2SO_4 by heating 24 hr at 80°C while bubbling preheated air through the solution; copper deposited electrolytically from 95 ml of the solution at a current density of about 10 amp/ ft^2 and a cell potential of about 2 volts; cell temperature was 55°C .

Dissolution of Cement Copper

^{106}Ru dissolved, %	66
^{106}Ru in sludge, %	34

Electrowinning of Copper from $\text{CuSO}_4\text{-H}_2\text{SO}_4$ Solution

^{106}Ru on cathode, counts min^{-1} per g of Cu	1.1×10^4
Cu deposited, %	12.7
^{106}Ru deposited, %	0.32

2.2 Removal of Ruthenium from Cell Electrolytes

To minimize contamination of the electrolytic processing facility, some means of preventing excessive buildup of ruthenium in the electrolyte is needed. Previous attempts to remove ruthenium from the electrolyte by solvent extraction were not successful.²

In recent tests most of the ruthenium was removed from solutions of cement copper (25 g Cu and 50 g H_2SO_4 per liter, 1.2×10^4 counts min^{-1} ml^{-1} of ^{106}Ru) by adding sodium persulfate and heating at 80°C in a flask fitted with a reflux condenser, while bubbling a stream of preheated air through the solution (Table 2). Ruthenium was removed from the exiting air by passing it through caustic scrubbers. For heating times of 1 hr, the release of ruthenium from the solution increased from 31% at 40°C to 39% at 60°C and to 85% at 83°C . Heating at boiling for 15 min released about 80% of the ruthenium from the solution. With the latter conditions, decreasing the persulfate concentration from 0.04 to 0.02 M did not significantly change the results. Below 80°C , the released ruthenium deposited on the flask walls beneath the surface

Table 2. Removal of Ruthenium from Electrolyte Solution
by Oxidation with Sodium Persulfate

Procedure: $\text{Na}_2\text{S}_2\text{O}_8$ added to electrolyte solutions prepared by dissolving ^{106}Ru -contaminated cement copper in sulfuric acid; heated in flask fitted with a reflux condenser while bubbling preheated air through the solution; exiting air passed through two traps containing 1 M NaOH.

Temp. (°C)	Heating Time (min.)	$\text{Na}_2\text{S}_2\text{O}_8$ Conc. (M)	% of Initial ^{106}Ru			
			In Final Solution	Removed from Soln. (by Diff.)	In NaOH Trap	On Walls of Flask (by Diff.)
40	60	0.025	69	31	0	31 ^a
60	60	0.025	61	39	0	39 ^a
83	60	0.025	15	85	0	85 ^b
boiling	15	0.040	24	76	50	26 ^b
boiling	15	0.020	20	80	7	73 ^{b,c}

^a ^{106}Ru plated on flask; most of it was beneath the solution.

^b ^{106}Ru plated on flask; most of it was above the solution.

^c No flow of air in this test.

of the solution. At 80°C and above, most of it deposited above the surface of the solution. In the test at the boiling temperature, about 2/3 of the released ruthenium carried over to the NaOH trap. This carry-over decreased to about 10% in another test in which no air was passed through the system.

3. NUCLEAR EXPLOSIVES IN OIL SHALE PROCESSING

W. D. Arnold

The feasibility of recovering oil from shales by underground retorting is under study by several government agencies and commercial companies. A nuclear device could be used to crush the shale prior to retorting. We are studying the oil recovery system with regard to the behavior of the radioactive contaminants that would be formed in a nuclear detonation. The test results reported here were obtained from studies of 1) the leaching of test shot debris with shale oil and 2) the distillation of shale oil.

3.1 New Samples of Test Shot Debris and Shale Oil

The radioactive test shot debris used in a leaching test described earlier² was several years old and some of the radionuclides (e.g., ⁹⁵Zr-Nb) that would be present in the chimney at the time of oil recovery (1 yr or more after the shot) had decayed. A sample of debris, purportedly rubble, from a more recent test shot was obtained from LRL. This debris contained some glassy material, apparently puddle glass, that was more radioactive than the bulk of the material. Taking samples that analyzed the same radiochemically was difficult, probably because some of the samples contained more of the glass than others. Consequently, the rubble was separated from most of the glass by gently grinding and screening the debris until only the hard pieces of glass were left. The debris, as received, was used in the leaching tests described below but the separated rubble will be used in future tests. Radiochemical analyses of the original debris and of the separated fractions are given in Table 3. The dominant radionuclides in the original debris were ⁹⁵Zr-Nb, ¹⁰³Ru, and

Table 3. Radiochemical Analysis of
Test Shot Debris

Procedure: initial analysis was on a sample of debris, as received. The debris was separated into 34.3 g of rubble and 7.4 g of glass by screening.

Radionuclide	Concentration (dis min ⁻¹ g ⁻¹)		
	As-received Debris (4/17/67)	Separated Debris	
		Rubble (6/20/67)	Glass (6/20/67)
⁸⁹ Sr	2.8 x 10 ⁶	1.1 x 10 ⁶	1.5 x 10 ⁶
⁹⁵ Zr-Nb	9.3 x 10 ⁷	1.1 x 10 ⁷	4.7 x 10 ⁷
¹⁰³ Ru	1.9 x 10 ⁷	3.8 x 10 ⁶	1.3 x 10 ⁷
¹⁰⁶ Ru	a	≤ 4.0 x 10 ⁶	1.6 x 10 ⁷
¹³⁷ Cs	a	≤ 4.0 x 10 ⁵	≤ 3.0 x 10 ⁵
¹⁴⁰ Ba-La	2.8 x 10 ⁶	≤ 1.0 x 10 ⁵	≤ 5.0 x 10 ⁵
¹⁴¹ Ce	1.4 x 10 ⁷	4.1 x 10 ⁶	1.2 x 10 ⁷
¹⁴⁴ Ce	a	≤ 4.0 x 10 ⁷	2.5 x 10 ⁷
Gross γ ^b	~3.5 x 10 ⁷	2.2 x 10 ⁷	8.1 x 10 ⁷

^a not reported

^b Gross γ measured in counts min⁻¹ g⁻¹

^{141}Ce . The longer-lived radionuclides ^{106}Ru , ^{137}Cs , and ^{144}Ce were not detected in analysis of this sample, but were found in the later analyses of the separated glass and rubble. The glass was about 4 times more radioactive than the rubble. All of the analyses were made by scanning the gamma spectra of the samples and having the scans analyzed by a computer.

A 5-gallon sample of shale oil was obtained from the U. S. Bureau of Mines Petroleum Research Center at Laramie, Wyoming. It is a liquid at room temperature. About 9 v% water and about 7 v% of a heavy sludge settled out of the oil when it was warmed and allowed to stand for several days. The crude oil separated from the water and sludge by decantation had a specific gravity of 0.908 at 26°C.

3.2 Leaching of Test Shot Debris

Heating a 5.2 g sample of the debris (as received) for 4 hr at 220°C under reflux with 25 ml of shale oil (not separated from the water and sludge) resulted in contamination of the oil with about 1×10^5 counts $\text{min}^{-1} \text{ml}^{-1}$ of gamma activity. The debris was releached with two successive 25 ml portions of fresh oil, each time yielding oil contaminated to the same level as the first oil sample. The debris was then leached twice more, each time with 50 ml of oil. The gamma activity of the oil from these last two contacts was 5×10^4 counts $\text{min}^{-1} \text{ml}^{-1}$. The gross gamma activity of the total oil from the five leaches was equivalent to about 7.5% of the gross gamma activity of the debris sample. Most of the activity in the oil samples was due to $^{95}\text{Zr-Nb}$ and ^{103}Ru . A total of about 2% of the $^{95}\text{Zr-Nb}$ and 5% of the ^{103}Ru was leached. The first two oil samples contained about as much ^{141}Ce as ^{103}Ru and smaller concentrations of $^{140}\text{Ba-La}$. All of the radioisotopes detected in the oil have relatively short half-lives (65 days or less) and their importance would be much less in an actual shale oil recovery operation (assuming start of retorting at 1 yr or more after the shot) than in these tests with debris which was only a few months old. Longer-lived radionuclides such as ^{106}Ru , ^{137}Cs , and ^{144}Ce that would be of greater importance in longer-cooled material were not detected in these tests

Table 4. Leaching Test Shot Debris with Shale Oil

Procedure: 5.2 g of as-received test shot debris (analysis in Table 1) was heated (under reflux but not stirred) 4 hr at 220°C with successive samples of fresh shale oil; oil was decanted, centrifuged, and sampled.

Leach No.	Volume of Oil (ml)	Radioactivity of the Oil									
		Gross γ		$^{95}\text{Zr-Nb}$		^{103}Ru		^{141}Ce		$^{140}\text{Ba-La}$	
		conc. ^a	% of Total	Conc. ^b	% of Total $^{95}\text{Zr-Nb}$	Conc. ^b	% of Total ^{103}Ru	Conc. ^b	% of Total ^{141}Ce	Conc. ^b	% of Total $^{140}\text{Ba-La}$
1	25	1.0×10^5	1.4	5.7×10^4	0.30	3.8×10^4	1.0	2.9×10^4	1.0	0.46×10^4	0.8
2	25	1.3×10^5	1.8	11.2×10^4	0.58	3.4×10^4	0.9	3.1×10^4	1.1	0.15×10^4	0.3
3	25	1.1×10^5	1.5	8.7×10^4	0.45	3.6×10^4	0.9	c	-	d	-
4	50	0.50×10^5	1.4	3.3×10^4	0.34	1.9×10^4	1.0	c	-	d	-
5	50	0.54×10^5	1.5	3.5×10^4	0.36	2.7×10^4	1.4	c	-	d	-

^a Counts min⁻¹ ml⁻¹

^b Dis min⁻¹ ml⁻¹

^c The oil contained some ^{141}Ce but the concentration was too low to measure accurately.

^d Not detected

because of interference with radiometric analyses due to the high concentration of the shorter-lived radionuclides.

In two other tests at lower temperatures and a lower debris/oil ratio, increasing the temperature from 103 to 196°C increased the amount of radionuclides leached by about 50%:

Temperature (°C)	Gross γ Activity of Oil	
	Counts min ⁻¹ ml ⁻¹	% of total
103	8.5×10^3	2.4
196	13.9×10^3	3.9

In these tests, 100 ml samples of the shale oil were heated under reflux for 5 hr with 1 gram samples of debris while stirring with a magnetic stirrer. The activity of the oil increased throughout the heating period but had about leveled-off at 5 hr.

3.3 Distillation of Shale Oil

The U. S. Bureau of Mines Petroleum Research Institute at Laramie, Wyoming was visited by W. D. Arnold in May, 1967, for discussions on refining crude shale oil. Much helpful information was obtained concerning laboratory equipment and techniques used in studying shale oil.

Crude shale oils are characterized by distilling them into four fractions:^{3,4}

- 1) Naphtha, boiling to 200°C at atmospheric pressure;
- 2) light oil, boiling to 225°C at 40 mm Hg;
- 3) heavy oil, boiling from 225°C to 300°C at 40 mm Hg; and
- 4) residuum.

We plan to fractionate samples of the radiocontaminated shale oil in this manner in order to follow the paths of the radionuclides in the distillation process. After sufficient volumes of the various fractions have been collected, means of removing the radionuclides from the contaminated fractions can be studied.

A distillation unit was designed and built. In the first run (with uncontaminated crude oil), the products were 0.9 v% naphtha, 23.5 v% light oil, 24.8 v% heavy oil, and 41.0 v% residuum. The material

balance was 90%. The column temperature could not be controlled to the extent desired during this distillation and some modifications are being made to improve the performance.

4. THERMAL ANALYSIS OF ACTINIDES

Work on the thermal analysis of actinide elements was extended to americium compounds during this report period. Americium oxide, americium oxalate, americium Sol-Gel material, and some solid complexes of americium were analyzed at temperatures up to 1600°C. The studies were interrupted when a glass component of the vacuum system failed, resulting in damage to the balance. The studies with americium will continue when the balance is again operable.

The micro-thermobalance and the separate differential thermal analysis unit previously described are nearly complete and cold testing of the two units will begin in the immediate future.

5. REPORTS ISSUED

1. C. A. Blake, Jr., and D. J. Crouse, Chemical Applications of Nuclear Explosions (CANE): Progress Report for January 1 to March 31, 1967, ORNL-TM-1886.
2. Other progress reports in this series:

ORNL-TM-1775	October 1 - December 31, 1966
ORNL-TM-1688	July 1 - September 30, 1966
ORNL-TM-1582	April 1 - June 30, 1966
ORNL-TM-1496	January 1 - March 31, 1966
ORNL-TM-1394	October 1 - December 31, 1965
ORNL-TM-1275	July 1 - September 30, 1965
ORNL-TM-1191	January 1 - June 30, 1965
ORNL-TM-1040	October 1 - December 31, 1964
ORNL-TM-963	July 1 - September 30, 1964

6. TALKS PRESENTED

J. W. Landry, "Quick and Dirt-Cheap: Projects from Peaceful Nuclear Explosives," presented to the dinner meeting of the QAD Seminar-Workshop conducted by the Radiation Shielding Information Center ORNL and LASL, Oak Ridge, Tennessee, April 17, 1967.

J. W. Landry, "Peaceful Uses of Nuclear Explosives, or How I Learned to Love the H Bomb," presented to the dinner meeting of the Civil Defense Research Project Annual Information Meeting, Oak Ridge, Tennessee, April 25, 1967.

J. W. Landry, "Canals and Other Colossal Construction with Nuclear Explosives," presented to the U. S. Navy Research Reserve Nuclear Science Seminar, Oak Ridge, Tennessee, May 21, 1967.

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2. Chemical Applications of Nuclear Explosives (CANE): Progress Report for January 1 to March 31, 1967, ORNL-TM-1886.
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4. G. U. Dinneen, Petroleum Refiner 33, 113 (1954).

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