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CHEMICAL APPLICATIONS OF NUCLEAR EXPLOSIONS (CANE):
PROGRESS REPORT FOR JANUARY 1 TO MARCH 31, 1967

Compiled by:

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ABSTRACT

In studies of variables affecting ^{106}Ru contamination of cement copper, little change in the amount of contamination was observed with change in agitation rate during cementation. Attempts to preferentially dissolve ^{106}Ru from cement copper were not successful. Of eleven radionuclides tested, only $^{95}\text{Zr-Nb}$, ^{59}Fe , and ^{110}Ag were extracted significantly by the copper extractant, LIX-64. Since the concentrations of these radioisotopes in the leach liquor are expected to be very low, solvent extraction of copper with LIX-64 should produce a copper product solution essentially free of radionuclides.

Contacting hot shale oil with radioactive debris from an underground test shot contaminated the oil with radionuclides. The principal contaminant was ^{106}Ru .

The measured rate of tritium-hydrogen exchange in water vapor-methane systems in a 1-liter glass vessel at room temperature and atmospheric pressure is

$$R \sim 2.3 \times 10^{-8} [\text{HTO}]^2 \text{ mc/liter-day}$$

when the tritium concentration is in the range of 10 to 50 mc/liter.

A study is being made of the thermal stability of various actinide compounds which might be of interest in transplutonium element production with thermonuclear devices. Thermogravimetric and differential thermal analysis equipment has been installed in a glove-box facility. Operability of the equipment has been demonstrated successfully in studies on europium and plutonium oxides.

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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, (3) the measurement of rates of exchange of tritium and hydrogen in water and natural gas under conditions which will aid the feasibility studies of the stimulation of gas production from wells by nuclear explosives (Project Gasbuggy), and (4) thermal analysis of actinide compounds.

Studies of hypervelocity jet sampling as a means of removing a specimen after irradiation in the neutron flux of a detonation were not active this quarter. This project is complete except for analysis of the amount of gold in the wood used to catch the jetted targets in the 5 final tests at the Vincentown, N. J. test range.

2. NUCLEAR EXPLOSIVES IN COPPER ORE PROCESSING

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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. Included in this report are the results of tests studying (1) ^{106}Ru contamination of cement copper, and (2) solvent extraction as a possible alternate method for copper recovery.

2.1 Ruthenium Contamination of Cement Copper

As shown in earlier tests,¹⁻³ ^{106}Ru is the only radionuclide that appears important with respect to radiocontamination of the cement copper products. Ruthenium and copper are not separated when the cement copper is smelted into a consumable anode, but they are efficiently separated during electrolytic purification of the anode.³ Some method for decreasing ruthenium contamination of the cement copper is needed, however, to minimize contamination of the electrolytic processing facility.

2.1.1 Effect of Agitation Rate on Copper and Ruthenium Cementation

Changing the stirrer speed in the range of 400-700 rpm had no effect on the amount of ruthenium contamination in cement copper products prepared by stirring synthetic leach liquors with pieces of detinned cans (Table 1). The amounts of ruthenium and copper cemented in 30 min were about 15% and 51-57%, respectively, for stirrer speeds in this range. With a stirring speed of 250 rpm, less ruthenium was cemented and copper recovery was higher (65%) so that the ruthenium concentration in the cement copper was only about 65% of that obtained at the higher stirring speeds. The observed decrease in recovery of copper with increase in the amount of agitation is contrary to results reported in the literature and is being checked.

2.1.2 Washing of Cement Copper

Attempts to preferentially dissolve ruthenium from contaminated cement copper were not successful. Some ruthenium dissolved in 1 M NaOH and 1 M HCl, but part of the copper also dissolved in these reagents:

<u>Weight of Cement Copper (g)</u>	<u>Solution</u>	<u>Solution Volume (ml)</u>	<u>^{106}Ru Dissolved (%)</u>	<u>Cu Dissolved (%)</u>
0.7	1 <u>M</u> NaOH	5	9	3
0.7	1 <u>M</u> HCl	5	22	10
0.7	6% H_2O_2	10	<1	--

Table 1. Effect of Stirring Speed on Ruthenium Contamination of Cement Copper

Procedure: 200 ml of synthetic copper ore leach liquor (2.0 g Cu^{2+} , 1.0 g Fe^{3+} , 3.0 g Fe^{2+} , 0.5 g NaCl, 0.4 g Na_2SO_4 per liter, about 2000 counts $\text{min}^{-1} \text{ ml}^{-1}$ of ^{106}Ru , pH 2.0) stirred for 30 min under a nitrogen atmosphere with 0.7 g of detinned cans; the amounts of ruthenium and copper cemented were calculated from analyses of the head liquor and supernate.

Stirrer Speed (rpm)	Cu Cemented (%)	Ru Cemented (%)	Ru in Cement Copper (counts $\text{min}^{-1} \text{ g}^{-1}$)
250	65.0	12.0	1.9×10^5
400	54.8	14.7	2.9×10^5
550	56.8	16.6	2.9×10^5
700	51.3	15.1	2.9×10^5

Ruthenium was not dissolved by 6% H_2O_2 . These tests were made by shaking samples of ruthenium-contaminated cement copper with the solutions for 5 min and analyzing the supernate for ruthenium and copper.

2.1.3 Extraction of Ruthenium from Cell Electrolyte

During electrolytic purification of the smelted copper anodes containing ^{106}Ru , most of the ^{106}Ru was dissolved in the cell electrolyte.³ Some means of preventing excessive buildup of ruthenium in the electrolyte is needed.

Attempts to remove ruthenium from a synthetic cell electrolyte by solvent extraction were not very promising with the eleven extractants tried (Table 2). These included amines, organophosphorous acids and esters, carboxylic acids, and sulfonic acids. Best results were obtained with 0.1 M solutions of Primene JM and Aliquat 336 in varsol, which extracted about 20% of the ruthenium in contacts at an organic/aqueous phase ratio of 1/1 from freshly prepared electrolyte solution. However, the extraction efficiency decreased with aging of the synthetic electrolyte solution. Measuring the age of the solution from the time the ruthenium was added, the ruthenium extraction with Primene JM dropped from 21% at 1 hr to 5.7% at 5 days and 5.2% at 6 days. The extraction with Aliquat 336 dropped from 17.3% at 1 hr to 6.9% at 6 days. With an electrolyte aged 5 days, a total of only 15% of the ruthenium was extracted when the electrolyte was

Table 2. Extraction of Ruthenium from Electrolyte

Procedure: synthetic electrolyte solution (45 g Cu and 200 g H₂SO₄ per liter, 3×10^4 counts min⁻¹ ml⁻¹ of ¹⁰⁶Ru) was contacted for 10 min at a 1/1 phase ratio with the solvents listed below and the ¹⁰⁶Ru concentration in the organic and aqueous phases was measured. The electrolyte age is measured from the time the ruthenium was added.

Organic Phase	% of ¹⁰⁶ Ru extracted from electrolyte aged		
	1 hr	5 days	6 days
0.1 M Primene JM in varsol	21.0	5.7	5.2
0.1 M Amberlite LA-1 in varsol	8.0	-	3.4
0.1 M Alamine 336 in 97% varsol--3% tridecanol	11.3	-	4.?
0.1 M Aliquat 336 in 97% varsol--3% tridecanol	17.3	-	6.9
1.0 M tributyl phosphate in Amsco 123-15	0.9	-	1.0
1.0 M diamylamyl phosphonate in Amsco 123-15	6.0	-	3.3
0.1 M di(2-ethylhexyl)phosphoric acid in varsol	<0.1	-	-
0.25 M Sunaptic Acid A in Amsco 125-82	<0.1	-	-
0.1 M neotridecanoic acid in varsol	<0.1	-	-
0.2 M dinonylnaphthalene sulfonic acid in varsol	<0.1	-	-

cascaded against 5 successive volumes of 0.1 M Aliquat 336 solution (all contacts at a phase ratio of 1/1).

2.2 Solvent Extraction of Copper

Recovery of copper from the leach liquor by solvent extraction with LIX-64 is a potential alternative to the cementation process.^{3,4} The extracted copper can be stripped from the solvent with sulfuric acid and fed directly to the electrolysis operation.

The relative extractibilities of copper and some of the radionuclides that could be present in the copper recovery system were measured between pH 1.5 and 2.5, the pH range of interest in copper ore processing. In 5-min extractions with 10 ^{w/v}% LIX-64 in varsol from synthetic leach liquors containing 0.2 g of Cu per liter and tracer amounts of the radiocontaminants, the extraction coefficient for copper was 10 at pH 1.6 and greater than 50 at pH 1.9 and above. Ruthenium-106, the most troublesome radiocontaminant in the cementation process, had extraction coefficients of less than 0.005 over the pH range, as did ¹³⁴Cs, ⁸⁵Sr, ⁶⁰Co, ⁴⁸Sc, ⁶⁵Zn, and ¹⁴⁴Ce. Only three radionuclides (⁹⁵Zr-Nb, ¹¹⁰Ag, ⁵⁹Fe) of the eleven tested had extraction coefficients greater than 0.01 (Fig. 1). The ⁹⁵Zr-Nb extraction coefficient decreased from 2 at pH 1.5 to 0.9 at pH 2.4. The coefficients for ¹¹⁰Ag and ⁵⁹Fe increased with increasing pH, reaching maximum values of about 0.6 and 0.1, respectively.

Further study of the extraction of ⁹⁵Zr-Nb, ⁵⁹Fe, and ¹¹⁰Ag showed extractions of the first two to be time dependent (Fig. 2). In extractions of ⁹⁵Zr-Nb from solutions initially at pH's of 2.7 and 1.5, the extraction coefficients increased with increasing contact time to values greater than 50 at 1 hr. No iron was extracted from the more acidic solution in 1 hr but, from the solution initially at pH 2.7, iron extraction coefficients increased from 0.01 at 5 min to 0.1 at 15 min and 0.15 at 1 hr.

Essentially all of the extracted ⁵⁹Fe but only about 30% of the ¹¹⁰Ag and 20% of the ⁹⁵Zr-Nb were stripped from the extracts with 2 M H₂SO₄ in a 10-min contact at a phase ratio of 1/1.

Fortunately, it is anticipated that the concentrations of ⁹⁵Zr-Nb, ⁵⁹Fe, and ¹¹⁰Ag in the leach liquors obtained by in-situ leaching of nuclear-broken ore will be very low. Solvent extraction with LIX-64, therefore, should produce a copper product solution essentially free of radionuclides.

ORNL DWG 67-5275

Fig. 1. Effect of pH on Extraction of Radioactive Contaminants with LIX-64. Organic phase: 10 ^{w/v} % LIX-64 in varsol. Aqueous phase: synthetic copper ore leach liquor containing 0.1 M SO_4^{2-} and, in g/liter, 0.2 Cu, 1.0 Fe^{3+} , 3.0 Fe^{2+} , and 0.5 NaCl; traced with the radioactive contaminants. Contact: 5 min at phase ratio of 1/1. The extraction coefficient for copper was 10 at pH 1.6 and >50 at pH 1.9 and above. Radionuclides which had extraction coefficients less than 0.005 were ^{106}Ru , ^{134}Cs , ^{85}Sr , ^{144}Ce , ^{60}Co , ^{65}Zn , ^{46}Sc and ^3H .

ORNL DWG 67-5276

Fig. 2. Effect of Contact Time on Extraction of Radionuclides with LIX-64. Organic phase: 10 ^{w/v} % LIX-64 in varcol. Aqueous phase: synthetic copper ore leach liquor (composition in Fig. 1) traced with the radionuclides. Phase ratio: 1/1. Temp.: ~23°C. Negligible amounts of ^{59}Fe were extracted from the pH 1.5 solution in 1 hr; extractions of ^{110}Ag from this solution were not measured.

3. DISTRIBUTION AND POSSIBLE FATE OF RADIONUCLIDES IN RECOVERING OIL FROM SHALES

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C. A. Blake, Jr.
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Conceivably, the product shale oil can be contaminated with radionuclides at several points in the process cycle, for example:

1. Exchange of tritium and hydrogen between water and shale oil during the retorting phase.
2. Dissolution of oil-soluble fission product compounds present in the shale rubble and puddle glass.
3. Inclusion of tritiated hydrocarbons which may be produced in the fireball and which will be dispersed in the chimney.

Additionally, the gases emerging from the retort will be contaminated with tritium and ^{85}Kr .

The relative amounts of the various fission products and tritium in the system will depend, of course, on the type of device used and the elapsed time following the shot. Assuming use of a predominantly fusion device and a wait of at least a year prior to the start of retorting, tritium will be by far the predominant radionuclide present. Most of the fission products and induced radionuclides produced will be trapped in the fused rock (puddle glass) that accumulates at the bottom of the nuclear chimney. The crushed shale, however, will be contaminated with fusion product tritium (presumably as tritiated water), fission products having gaseous precursors (for example ^{90}Sr , ^{137}Cs , ^{91}Y), and volatile ^{106}Ru . In addition the void space in the chimney will contain krypton and tritium (assumed to be mostly tritiated water vapor).

Short of an actual nuclear test, it is impossible to assess accurately the potential problems involved in producing and handling the oil. To obtain some understanding of the type and degree of radiocontamination of the oil that may occur, we are retorting shale spiked with radionuclides in small-scale equipment and are leaching nuclear test shot debris with shale oil.

3.1 Leaching of Test Shot Debris

Rubble and puddle glass from an underground test shot were heated with

samples of shale oil (produced in our laboratory retort) in a flask fitted with a reflux condenser. After 4 hr at 220°C, the oil contained about 0.2% of the gross gamma activity initially present in the rubble and about 0.02% of that in the puddle glass (Table 3). More than 85% of the gamma activity of the oil that was heated with the rubble was due to ^{106}Ru and about 10% was due to ^{137}Cs . The gamma activity of the oil sample that was heated with the puddle glass was mostly from ^{106}Ru (56%) and ^{60}Co (42%). About 1.5% of the ^{90}Sr was dissolved from the rubble and less than 0.01% from the glass.

The test shot debris used in these tests was several years old and certain fission products, e.g., $^{95}\text{Zr-Nb}$, ^{91}Y , etc., that would be present in nuclear-broken shale at the time of retorting had decayed almost completely. A sample of rubble from a recent test shot has been obtained from LRL for studying the behavior of the shorter-lived radionuclides.

Results of these preliminary tests and the laboratory retorting test with shale spiked with tritiated water reported previously⁵ indicate that the crude oil from nuclear-broken shale will probably be contaminated, but to an unknown extent, with tritium and certain fission products. Consequently, consideration is being given to methods for minimizing contamination of the oil and methods for decontaminating the oil both at the site and in subsequent refining operations.

4. TRITIUM EXCHANGE IN NATURAL GAS-WATER SYSTEMS

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J. M. Schmitt

In feasibility studies of the stimulation of gas production from wells by nuclear devices (Project Gasbuggy), it is important to consider the relative distributions of tritium gas (produced by the device) to the groundwater and to the natural gas (e.g., methane) initially present in the cavity, and to the gas that enters the well once gas production has started. Because of the extreme temperatures and pressures at the instant of detonation and shortly thereafter, the tritium distribution occurring during this phase can be measured realistically only in an actual nuclear test. However, laboratory measurements have been made which provide an indication of the tritium exchange to be expected during the gas production phase.

Table 3. Contamination of Shale Oil with Radionuclides from Test Shot Debris

Procedure: 10 ml samples of shale oil were heated 4 hr at 220°C with 10 g samples of debris from an underground nuclear test shot. The oil was decanted, centrifuged, and the supernate analyzed.

Radionuclide	Leaching of Rubble			Leaching of Glass		
	Total Activity of Initial Solids (dpm) ^a	Total Activity of Oil (dpm)	Amount Dissolved (%)	Total Activity of Initial Solids (dpm) ^a	Total Activity of Oil (dpm)	Amount Dissolved (%)
Gross γ	6.8×10^4	129 ^a	0.2	6.8×10^5	138 ^a	0.02
⁶⁰ Co	810	10	1.5	3.0×10^5	224	0.08
⁹⁰ Sr	8.6×10^3	128	1.5	3.9×10^5	38	<0.01
¹⁰⁶ Ru	3.9×10^3	540	13.9	6.5×10^4	295	0.5
¹³⁷ Cs	2.1×10^5	66	0.03	1.3×10^5	<10	<0.01
¹⁴⁴ Ce	940	14	1.5	5.2×10^5	<10	<0.002

^aGross gamma activity measured in counts per minute.

Such tests have shown that the formation of tritiated methane is probably low, provided that the tritium concentration in the gas phase is low and provided that there is no catalytic effect from the materials present in the cavity. Specifically, the rate of tritium exchange with hydrogen in methane-water vapor mixtures at room temperature and atmospheric pressure in a 1-liter glass vessel has been found to be

$$R \sim 2.3 \times 10^{-8} [\text{HTO}]^2$$

where R is the rate of exchange in mc/liter-day and the tritium (as HTO) concentration is in the range of 10 to 50 mc/liter. In a test performed last year at 100°C the exchange rate was equal to or less than 2.2×10^{-6} mc/liter-day when the tritium concentration was 1 mc/liter.

It is possible that the exchange rates within the nuclear cavity can be different from those reported above as a result of the presence of the large surface area of the crushed rock within the cavity. Laboratory experiments with crushed shale (from the gas-producing geological formation) in the exchange vessel were inconclusive. Although the shale had been exposed to water vapor for several days prior to its addition to the tritiated water-methane mixture, a large portion of the tritiated water was absorbed by the shale as the experiment progressed. Within 30 days, for example, the gas phase tritium concentration decreased from 20 to 2 mc/liter. The measured exchange of tritium with hydrogen in the methane during the same period was about the same as if all the tritium had remained in the gas phase.

5. THERMAL ANALYSIS OF ACTINIDE COMPOUNDS

The interest in producing transplutonium elements by irradiating targets in the high neutron flux of thermonuclear devices has encouraged

investigations of the thermal stability of various actinide compounds.

The thermogravimetric analysis (TGA) and the differential thermal analysis (DTA) system to be used for studying actinide compounds has been described previously.³ Reported are equipment shakedown studies with europium oxide and initial tests with plutonium oxide.

5.1 Thermal Analysis of Europium Oxide

The equipment was tested with europium oxide microspheres prepared by the Sol-gel Process.⁶ Oxides known to contain water and small amounts of nitrate gave rapid weight loss at 250-300°C accompanied by a sharp endothermic DTA peak. The weight change corresponded to a loss of one mole of water per mole of europium and indicated an empirical formula $\text{Eu}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$. Weight losses and exothermic peaks in the 350-400°C region have been tentatively ascribed to nitrate decomposition.

5.2 Thermal Analysis of Plutonium Oxides

After alpha containment of the TGA-DTA System was complete, the equipment was used to examine several samples of plutonium oxide. One sample known to contain nitrate was studied in vacuum, argon and oxygen atmospheres up to 1600°C. The patterns did not vary with atmosphere and each consisted of two weight loss steps merging at 150°C and indicating a 32% loss in weight. There were corresponding endothermic DTA peaks at 150°C and 270°C. The decomposition mechanism has not been completely elucidated; however, nitrate is lost in both steps. Decomposition products formed in the first weight loss period appear to consist of water and oxides of nitrogen, while the second weight loss corresponds to the measured loss in nitrate. A low-nitrate plutonia showed similar endothermic peaks, but only a small weight loss over the same temperature range. There were no detectable TGA or DTA changes in the 1200 to 1600°C range. X-ray analysis of all products showed the material to be PuO_2 .

6. REPORTS ISSUED

1. C. A. Blake, Jr., K. B. Brown, and D. J. Crouse, Chemical Applications of Nuclear Explosions (CANE): Progress Report for October 1 to December 31, 1966, ORNL-TM-1775.

2. Other progress reports in this series:

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

7. TALKS PRESENTED

J. W. Landry, "Engineers are Thinking Bigger with Nuclear Explosives," presented under the ORNL Traveling Lecture Program to the College of Engineering Seminar, University of South Carolina, January 5, 1967.

J. W. Landry, "Project Earth Furnace - Industrial Chemicals from Peaceful Nuclear Explosives," presented under the ORNL Traveling Lecture Program to the Student Chapter of American Institute of Chemical Engineers, University of Mississippi, February 6, 1967.

J. W. Landry, "Physicists Provide Potent Packages of Power for Progress - Peaceful Nuclear Explosives," presented under the ORNL Traveling Lecture Program to the Physics Department Seminar, University of Southern Mississippi, February 7, 1967.

J. W. Landry, "Scientific Applications of Nuclear Explosives," presented under the ORNL Traveling Lecture Program to the Physics and Nuclear Engineering classes at the University of Southern Mississippi, February 8, 1967.

J. W. Landry, "Chemical Applications of Nuclear Explosives," presented to the Oak Ridge High School Chemiteers, Oak Ridge, Tennessee, March 7, 1967.

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4. Chemical Applications of Nuclear Explosives (CANE): Progress Report for April 1 to June 30, 1966, ORNL-TM-1582.
5. Chemical Applications of Nuclear Explosives (CANE): Progress Report for October 1 to December 31, 1966, ORNL-TM-1775.
6. C. J. Hardy, S. R. Buxton, and M. H. Lloyd, Preparation of Lanthanide Oxide Microspheres by Sol-Gel Methods, ORNL-4000 (in progress).

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