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ULTRASONIC LEACHING OF URANIA-IMPREGNATED  
GRAPHITE FUELS

by

William B. Tarpley  
Robert S. Winchester

**AEROPROJECTS INCORPORATED**  
WEST CHESTER, PENNSYLVANIA

Contract No. AT (30-1)-1836

April 1961

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ULTRASONIC LEACHING OF URANIA-IMPREGNATED GRAPHITE FUELS

by

Aeroprojects Incorporated

ABSTRACT

The proposed use of unclad, urania-impregnated graphite fuel elements in experimental and power reactors raises the question of how best to reprocess the elements after irradiation. A grind-leach process using boiling nitric acid has been suggested as alternate to earlier combustion techniques. Besides requiring pre-grinding of the elements, a hazardous and difficult-to-control operation, the process did not leave a residue sufficiently low in radioactivity when the elements contained initially less than 3 w/o uranium.

Application of ultrasonic vibratory energy to the graphite during this process has increased leaching rate and completeness to such an extent that the practical process limit of 3% uranium is no longer a problem. The ultrasonic studies were carried out with non-irradiated specimen plates and ground pebble specimens. The plates apparently can be leached in practical time span to such a low level of radioactivity that disposal of the graphite residues is simplified. Furthermore it appears that the pre-grinding of the spent elements can be eliminated. Direct activation of the intact fuel plate specimens resulted in leaching efficiency at least as good as with ground specimens.

It is recommended that the studies be continued and enlarged to evaluate pilot plant processing problems that may be encountered with irradiated, spent elements. Other solid-liquid extraction and/or reaction processes should be examined for useful ultrasonic acceleration.

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ULTRASONIC LEACHING OF URANIA-IMPREGNATED GRAPHITE FUELS

I. INTRODUCTION

A. Reprocessing of Impregnated Graphite Fuels

The Philadelphia Electric (General Atomics) (1) and Sanderson and Porter (2) power reactors, and the Los Alamos (Turret) (3) experimental reactor are designed to operate with homogeneous urania-impregnated graphite fuel plates, pebbles and cylinders, respectively, in circulating inert gas coolants at elevated temperatures. These fuel elements are expected to give better neutron economy with higher gas and steam temperatures, and to result in lower fuel and capital costs than other types of reactors.

A satisfactory process for the recovery of fissionable material from the spent fuel elements, with efficient disposal of the waste and fission products, remains to be worked out. Suggested methods include (a) combustion of the spent elements in oxygen in a high temperature furnace, followed by dissolution of the residual metallic oxides in aqueous acid, and (b) leaching of the elements with nitric acid.

The combustion method has been investigated at ORNL and LASL with essentially complete recovery of uranium from non-irradiated material. However, this method presents both operational and disposal problems. Even careful regulation of the oxygen supply to the combustion furnace cannot prevent occasional carbon dust explosions, and many volatile radioactive substances, present in the spent fuel elements, will be released in the escaping gas giving a whole new set of decontamination problems.

Aqueous acid leaching methods have been investigated at ORNL (4) (5). These entail crushing and grinding the graphitic fuel elements, then treating them alternately with concentrated nitric acid and water for several cycles under reflux temperatures. Best recoveries were obtained from particles passing 200 mesh, and from materials containing more than 3% uranium.

Application of the grind-leach process to irradiated spent fuels will give rise to some acute handling problems. These have been anticipated by ORNL (4), but so far no solution has appeared more promising than remote-grinding, which is difficult to control and which carries with it the unavoidable dust-containment problem. Various methods for fracturing, disintegrating or grinding the graphite in the leaching solution itself, or in sealed vessels, were investigated; the only one which seemed

satisfactory was the fracturing of graphite pebbles in refluxing fuming nitric acid, but this limits the selection of vessel material. Titanium metal, which is satisfactory for most service with nitric acid, has been reported to give an explosion hazard after contact with refluxing fuming nitric acid (6).

The ORNL leaching process could be improved by the elimination of the separate grinding operation and by increasing the leaching efficiency to extend to concentrations of uranium below 3%. The ultimate goal is the reduction of radioactivity to a level sufficiently low in the carbon residue that it may be disposed of without further decontamination treatment. This report deals with the application of ultrasonic energizing techniques toward such improvements.

#### B. Ultrasonic Acceleration of Extraction, Diffusion and Dissolution

Recent work at Aeroprojects on the ultrasonic leaching of simulated calcined waste (7) showed that ultrasonic activation resulted in improved recoveries of the simulant fissionable products, in a small fraction of the time required for leaching without ultrasonics. This work also indicated that under optimum ultrasonic conditions leaching could be quantitative, leaving negligible radioactivity in the residual solids.

A literature search shows that ultrasonic treatment has extracted nucleoproteins from bakers' yeast (8), oils from fish materials (9), and protein from brewers' yeast (10) with greater efficiency than could be achieved by non-ultrasonic methods. Bitter materials have been extracted ultrasonically from hops on a commercial scale in a German brewery (11, 12). Ultrasonic processing for accelerated beet sugar recovery has been studied by Freeman (13). Thompson and Sutherland (14) have separated oil from peanuts with a 90% extraction efficiency in short-duration treatment.

Diffusion of NaCl and Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> through membranes can be accelerated 70-100% with ultrasonic vibration<sup>14</sup> (15, 16, 17). In research at Aeroprojects on the washing of photographic film, ultrasonic vibration has increased the removal of sodium thiosulfate (18). This laboratory has also demonstrated that the rate and quantity of sensitizer-ion transfer to the surfaces of AgBr particles of photoemulsions can be increased by ultrasonic application (19) without change in particle size. The effects were observed with 20-, 300-, and 800-kc activation.

Ultrasonic acceleration of diffusion has produced more rapid penetration of liquid carbon disulfide into an alkali cellulose matrix (20) and accelerated the transfer of phenanthrene from benzene to methyl alcohol (21).

In the dissolution of fuel elements, chemical reactions between the reagent and the metal often produce gases and/or corrosion products that are adsorbed on the metal surface. Ultrasonic activation promotes diffusion and reduces concentration gradients. In work at Aerojects (22) ultrasonic treatment has accelerated the dissolution of various corrosion-resistant metals. The ultrasonic dissolution of Ni in  $\text{HNO}_3$  has also been reported (23).

The exact mechanism by which such effects occur is controversial. Some investigators attribute the effects to changing pressures, accelerations, and temperature gradients in the sound field. Others believe that microagitation, radiation pressure, or cavitation is more significant. Tarnoczy (17) has decried efforts to explain ultrasonic diffusion phenomena in terms of any single mechanism and attempted, by controlled experimental conditions, to determine the effect attributable to each of several factors.

Alexander (24) postulated that ultrasonic diffusion effects achieved at solid-liquid interfaces are caused by reduction in resistance of the so-called Nernst diffusion layer of liquid next to the solid, and Nyborg (25) presented a plausible explanation for such a phenomenon in terms of the microstreaming which occurs in the vicinity of a bubble in a sound field, even in the absence of cavitation.

Many of the ultrasonic effects in liquid systems are attributed to the phenomenon of cavitation. The induction of suitably high internal tension ruptures the liquid at the sites of cavitation nuclei or weak spots in the cohesive structure of the liquid. These weak spots may consist of dissolved or entrained gases, impurities, dust particles, or other particulate matter. The bubbles formed during the tension phase of an elastic wave tend to collapse during the subsequent compression phase of the wave (26, 27).

Interfacial cavitation is responsible for destructive erosion, dispersion, ultrasonic cleaning of metal surfaces, and ultrasonic soldering. Cavitation effects of this type may be significant in the potential ultrasonic acceleration of the leaching of uranium from graphite fuel.

## II. EXPERIMENTAL ULTRASONIC LEACHING

### A. Approach to the Problem

The amount of experimental work that could be planned and carried out was limited by the nature and amounts of simulant materials which could be provided by ORNL. Accordingly, the ultrasonically-powered experimental apparatuses used were selected on the basis of past experience and, as they performed adequately, no attempts were made to introduce modifications or improvements, or to optimize the effects of different ultrasonic frequencies and power levels.

For the intact fuel plate specimens, experience (22) dictated that direct coupling of the specimen to the ultrasonic source would give best results. The granular specimens could not be coupled, but could be rested on the ultrasonically-activated bottom of a suitable vessel.

Chemical experimental conditions were selected, based on the work of Bradley and Ferris at ORNL (4), and the granular specimens corresponded to some that they had used.

## B. Experimental Equipment, Materials and Procedures

### 1. Equipment

Each of the two treatment arrays investigated comprised an ultrasonic transducer-coupling system and appropriate electronic or motor alternator driving equipment. Coupling members were designed for direct activation of the fuel plate samples and for bulk activation of the ground fuel in nitric acid solvent.

Each system operated at nominal 20 kc ultrasonic frequency and a power level of 300 watts. The direct coupler was made of stainless steel with a Nionel clamping fixture to securely hold the graphite plate specimen being activated (Fig. 1).

A cavity coupler made of titanium was used in the bulk activation of ground, sized, graphite fuel material (Fig. 2).

### 2. Materials

The simulant fuel materials provided by ORNL consisted of graphite plates (coded LCX-41), 1/4-inch thick, impregnated with 1.96% non-enriched uranium, and crushed graphite pebbles containing 2.0%, 4.2%, and 11.2% uranium, in sieve fractions of -16 + 30 mesh.

### 3. Analytical Procedure

The samples of graphite leach solutions taken at described intervals were analyzed for uranium content, using spectrophotometric methods (28, 29), in which measured aliquots were diluted with aluminum nitrate to give an acid deficient system and the uranium extracted into hexone. Uranyl-dibenzoyl-methane complex absorbancy was then measured at 410 m $\mu$ , using a model DU Beckman spectrophotometer. Standardization curves were made from the absorbancies of the uranyl-dibenzoylmethane complex from hexone extracts of aqueous uranyl solutions.

C. Ultrasonic Leaching Experiments

1. Leaching of Intact Fuel Plate Specimens

a. Effect of Nitric Acid Concentration

Specimen fuel plates (LCX-41, 1.96% uranium), each 2 x 2 x 0.25 inches and weighing approximately 20 grams, were immersed in refluxing nitric acid at 2, 4 and 10 M concentrations, in the apparatus shown in Figure 1. One set of samples was ultrasonically activated at a frequency of 20 kc and a power input of 300 watts, and a duplicate set was mechanically stirred without ultrasonics.

Aliquot samples of the leaching solution were taken at the stated intervals and analyzed for uranium content. The results are shown in Table I. Although ultrasonic leaching proceeded many times faster than non-ultrasonic leaching, it was evident that a higher concentration of nitric acid was desirable to rapidly effect complete leaching.

b. Effect of Time

Specimen fuel plates were set up as before, but in refluxing 15.7 M nitric acid. Aliquot samples of the leaching acid were removed at regular intervals and analyzed for uranium. At the end of 20 hours, the plates were ignited and the residue was analyzed for uranium. Results are shown in Table II and plotted as a graph in Figure 3.

Leaching was 99.8% complete after 20 hours with ultrasonic treatment, as compared with 92.2% in the non-ultrasonic control. Moreover, the rate curve (Figure 3) for the ultrasonic leaching showed no tendency to fall off; approximately 90% of the residual uranium was leached in each 7 hours of treatment. The non-ultrasonic rate was 2-1/2 times slower; 18 hours was required for 90% leaching.

2. Leaching of Ground, Sized Pebble Specimens

The sized (-16 + 30 mesh) material supplied by ORNL was treated by the following procedure: Approximately 10 grams were refluxed with 50 ml. of 15.7 M nitric acid for 4 hours in the titanium dissolver apparatus (Figure 2), under activation at 20 kc with ultrasonic power input of 300 watts. Non-ultrasonic controls were mechanically stirred under similar conditions. After the reflux, the supernatant liquid was removed by vacuum through a sintered glass immersion filter, and the residue was washed with three 25 ml. portions of hot water, each soaking for 15 minutes.



This procedure was repeated for a total of 2 to 4 cycles on each of the 3 samples (2.0, 4.2 and 11.2% uranium). The residual graphite was then dried, weighed and burned in air at 1100°C. Uranium determinations were made on the ash residue.

Results are listed in Table III, expressed in terms of percent uranium recovered. Better recoveries were obtained for all initial uranium concentrations with ultrasonic treatment than without. Moreover, the ultrasonic improvement was more pronounced for the lower uranium content specimen. For example, the average recovery from the sample containing the most uranium was 99.9% (ultrasonic) vs. 99.5% (non-ultrasonic); and, from the sample containing the least uranium, 99.5% vs. 94.0%.

### III. CONCLUSIONS AND RECOMMENDATIONS

#### 1. Conclusions

Direct application of ultrasonic energy to intact simulant uranium-impregnated graphite fuel plates permitted quantitative leaching of uranium within 20 hours, with no indication of reduction in leaching rate. To the extent these data can be applied to irradiated material, it would appear that, not only can the cumbersome grinding process be eliminated, but also the leaching can be carried to the degree where the residual graphite needs no further treatment for economic storage and disposal.

Ultrasonic leaching of the already-ground simulant pebble fuel was more efficient than non-ultrasonic leaching. From specimen material containing only 2% by weight of uranium, the uranium was 99.5% leached, as compared with 94.0% for a non-ultrasonic control and with 96.5% reported for similar material by ORNL (4).

#### 2. Recommendations

These experiments should be continued and expanded, with particular emphasis on the leaching of unground fuels. Intact pebble fuel was not available for the experiments reported here, but there is good reason to believe that it can be ultrasonically leached with adequate efficiency.

The emphasis on experimental data should be shifted away from the initial uranium concentration and the percent recovery (since 99.9% recoveries are in all instances possible) and focused on the absolute concentration of radioactive material remaining in the graphite after leaching, from the point of view of simplicity of disposal.

As soon as irradiated fuels become available, these studies should be immediately applied to them and work with non-irradiated fuels should be stopped, as physical changes during irradiation may have a great influence on the results.

Table I

**EFFECT OF ACID CONCENTRATION  
 ON ULTRASONIC LEACHING OF URANIUM  
 FROM URANIUM-GRAPHITE FUEL PLATE**  
 Fuel Plate: ORNL Code LCX-41, 1.96 w/o uranium

HNO <sub>3</sub> , M	Reflux Time, hr	Uranium Recovered, %	
		Ultra $\bar{s}$ onic	Non-ultrasonic
2	4	9.6	1.9
4	4	16.4	2.2
10	2	38.7	
10	4		26.1

Table II

**ULTRASONIC LEACHING OF URANIUM  
 FROM URANIUM-GRAPHITE FUEL PLATE  
 WITH 15.7 MOLAR NITRIC ACID**

Reflux Time, hr*	Uranium Recovered, %	
	Ultrasonic	Non-ultrasonic
4	76.8	
6	84.2	
8	95.2	
10	97.8	71.5
12	98.8	
16	99.2	
18	99.6	
20	99.8	92.2

\* Single run with leach solution analyzed at stated intervals.

Table III

ULTRASONIC LEACHING OF URANIUM-GRAPHITE  
FUEL SPECIMENS IN BOILING 15.7 MOLAR NITRIC ACID

Specimen Particle Size: -16 + 30 mesh  
Ultrasonic Frequency: 20 kc  
Ultrasonic Intensity: 300 watts

% Uranium In Sample	Type of Run	% Uranium Recovered				Uranium Concentration In Residue g/100 g Graphite
		First Leach	Second Leach	Third & Fourth Leach	Total* Recovery	
2.0	Non- Ultrasonic	87.9	2.8		90.7	0.18
		87.1	3.7		90.4	0.19
		85.8	3.9	3.7	93.4	0.13
		87.9	2.9	4.0	94.7	0.11
	Ultrasonic	90.7	4.3		95.1	0.10
		90.8	3.9		95.8	0.08
		91.3	4.1	4.2	99.6	0.01
		91.6	3.8	4.1	99.5	0.01
4.2	Non- Ultrasonic	98.3	0.7		99.0	0.04
		97.2	0.6		97.8	0.09
	Ultrasonic	99.1	0.4		99.6	0.017
		99.6	0.4		99.9	0.004
11.2**	Non- Ultrasonic	99.1	0.2		99.6	0.05
		99.3	0.1		99.4	0.07
	Ultrasonic	99.3	0.2		99.9	0.01
		99.6	0.2		99.9	0.01

\* By difference: 100% minus uranium found in residue.

\*\* These samples also contained 0.7% iron.

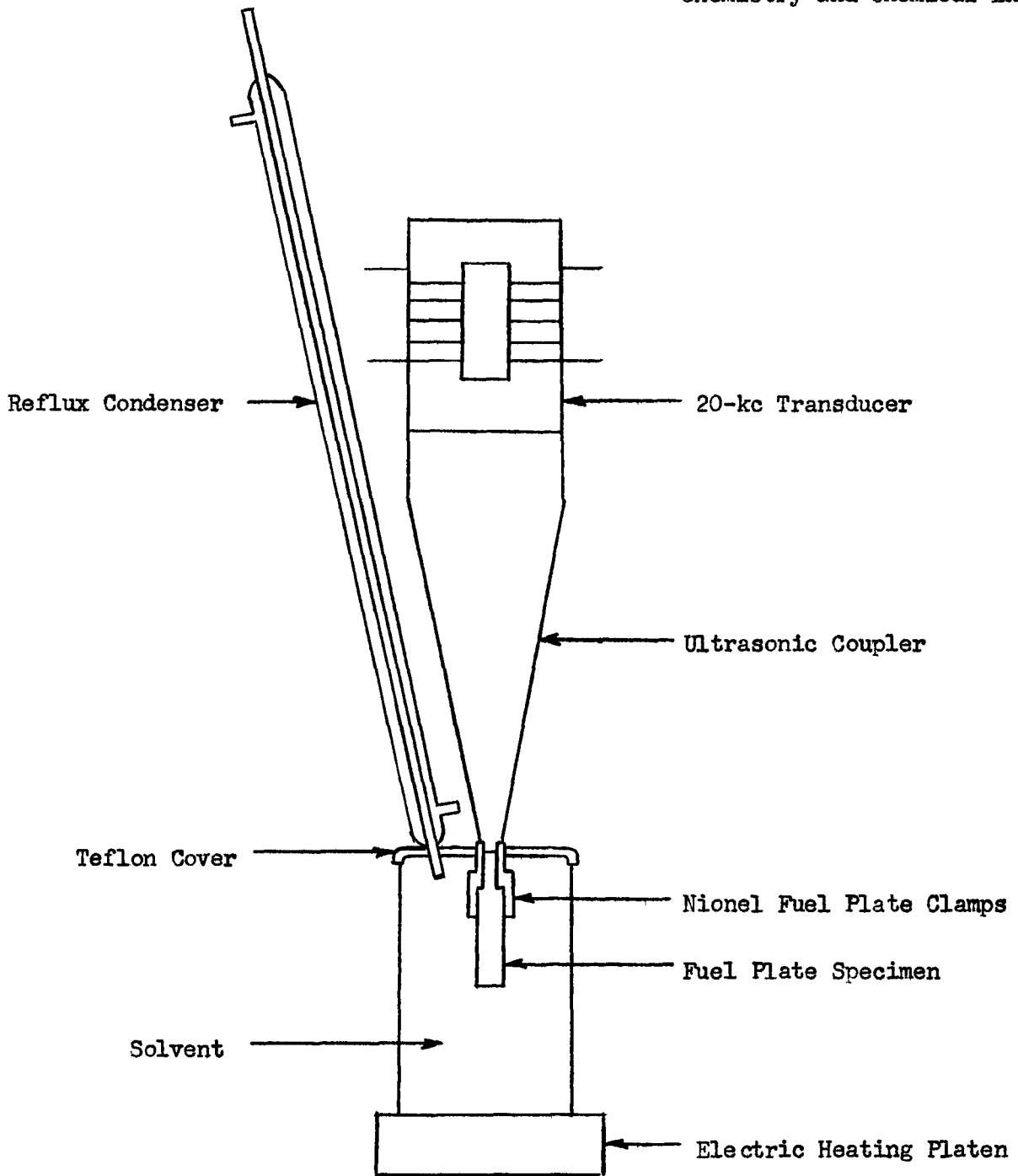


Fig. 1: DIRECT COUPLER ULTRASONIC LEACHING TEST ARRAY

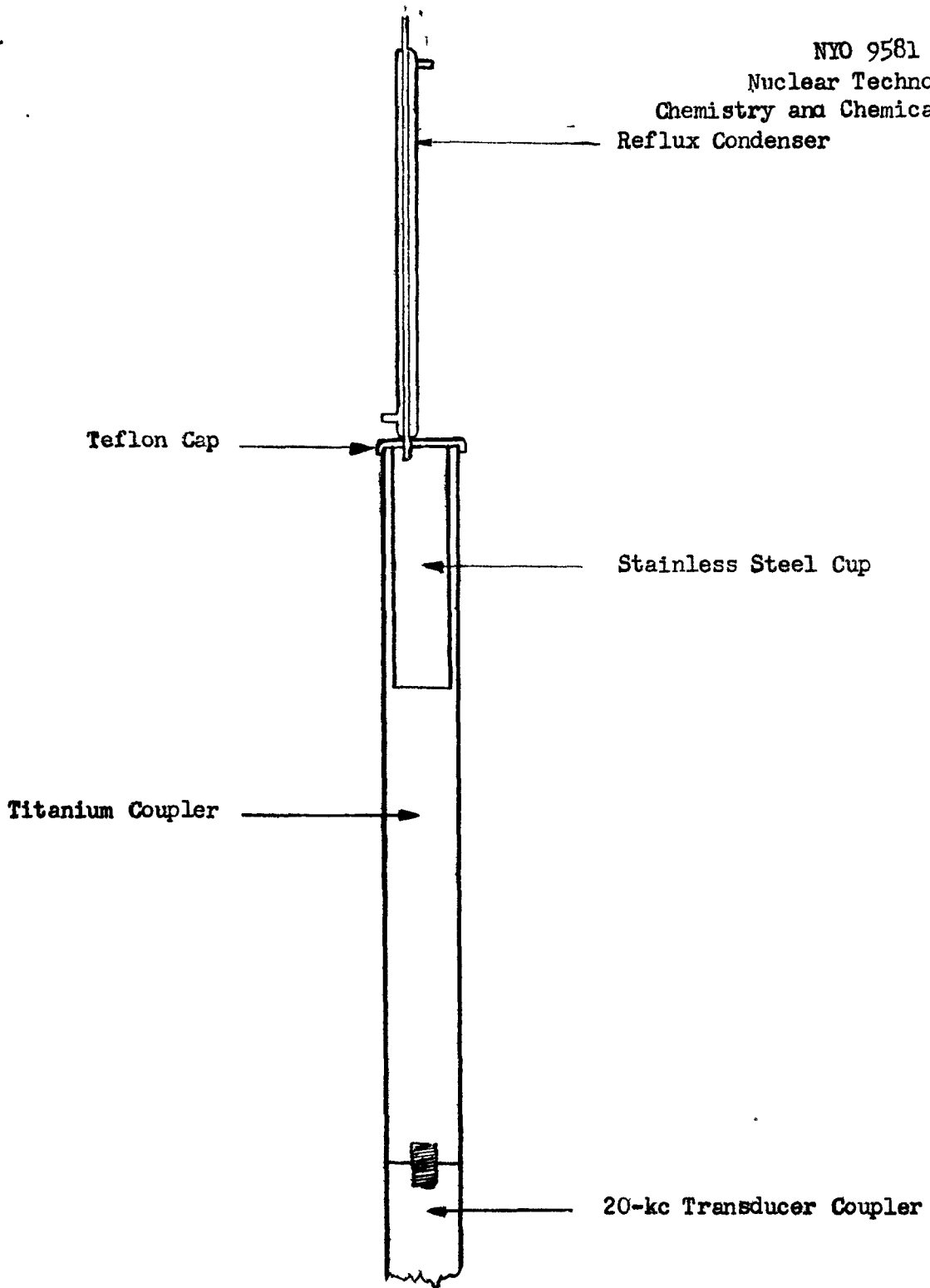
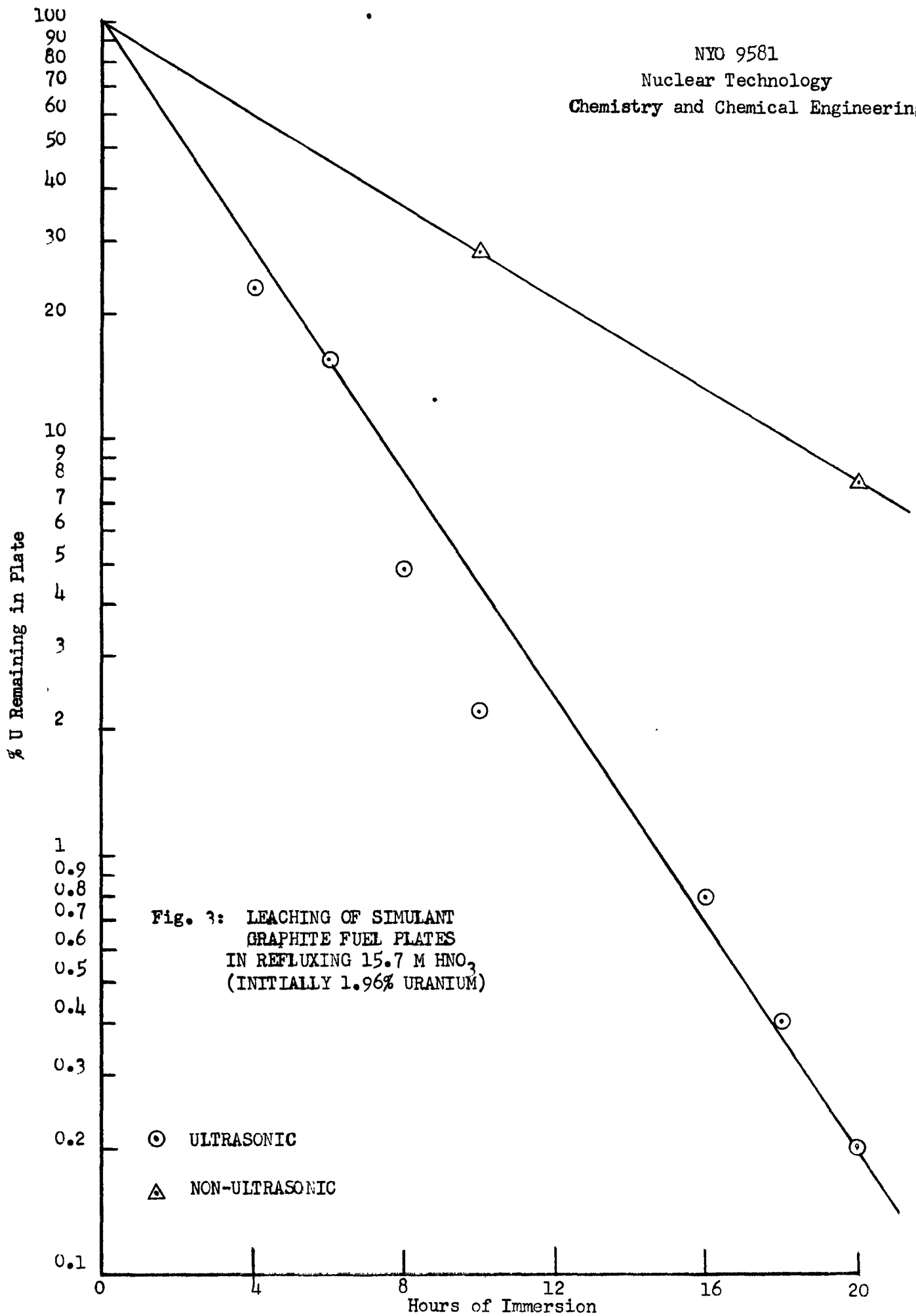


Fig. 2: ULTRASONIC CAVITY-COUPLER ARRAY WITH REFLUX CONDENSER



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