

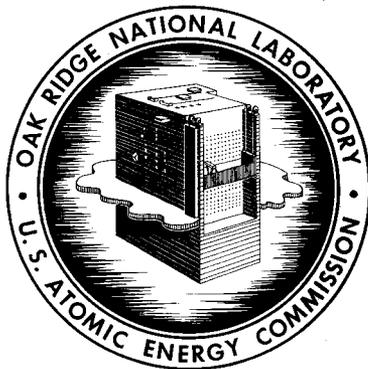


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ORNL-3023
UC-4 - Chemistry

CONSOLIDATED EDISON FUEL LOSSES
ON EXPOSURE TO IRRADIATED AND
AERATED SULFEX AND DAREX
DECLADDING SOLUTIONS

T. A. Gens



OAK RIDGE NATIONAL LABORATORY

operated by

UNION CARBIDE CORPORATION

for the

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ABSTRACT

Exposure of refluxing final Sulfex decladding solution (2.8 M sulfuric acid, 56 g of stainless steel per liter) to air while contacting nonirradiated Consolidated Edison type fuel pellets (about 6% uranium dioxide-94% thorium dioxide) caused total uranium losses to increase from 0.06 to 0.08%, a 30% increase, at 1 hr and to increase nearly 0.01% per hour thereafter. Similar treatment of the final Darex solution (2.9 M nitric + hydrochloric acid, 47 g of stainless steel per liter) caused total uranium losses to triple, from 0.25 to 0.90%, at 3 hr and increase 0.05% per hour thereafter. In contrast, losses caused by Co-60 radiations in the absence of air at a radiant power density of about 1 watt/liter were not large enough to measure accurately in refluxing final Darex solution, and were smaller than losses caused by aeration in refluxing final Sulfex solution. In refluxing initial Sulfex solution (6 M sulfuric acid), aeration and irradiation caused uranium losses to increase over 10-fold, from 0.017 to 0.29%, and 5-fold, from 0.017 to 0.086%, respectively, at 3 hr. In refluxing initial Darex solution (5 M nitric acid—2 M hydrochloric acid), aeration had a minor effect for the first 10 hr while irradiation caused uranium losses to double, from 0.15 to 0.30%.

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1.0 INTRODUCTION

This report describes laboratory-scale experiments made to determine if the uranium and thorium losses observed in decladding of Consolidated Edison type fuel (about 6% uranium dioxide-94% thorium dioxide) with Sulfex (1-3) or Darex (4-6) reagents are significantly altered by exposure of the fuel to air or irradiation at approximately the intensity expected in actual processing. The practicability of the flowsheets may depend on the effect of air and irradiation on the uranium, plutonium, or thorium losses. Hot-cell experiments similar to those described in this report are planned with neutron-irradiated pellets.

Uranium and thorium losses to decladding solutions of as high as 0.1% from dense Consolidated Edison type pellets (93% of theoretical density, the same as the actual fuel before irradiation) and as high as 0.4% from low density (80% of theoretical) pellets have been reported (7,8). Experiments performed in hot cells indicated that the presence of air caused larger uranium losses during decladding of stainless steel clad uranium oxide than did irradiation (9). Losses during both Sulfex and Darex decladding of irradiated Consolidated Edison fuel were comparable to losses with nonirradiated fuel in work done at Battelle Memorial Institute (10). Radiation-induced uranium losses during Sulfex decladding of stainless steel-clad uranium dioxide pellets have been found to be small at Hanford Atomic Products Operation (11).

The primary reaction that irradiation causes in dilute aqueous solutions, the formation of hydrogen and hydroxyl radicals, has been intensively investigated (12-14). Secondary reactions involving these radicals are known to produce stable products (hydrogen peroxide, Fe(III) from Fe(II), or H₂, for example) (13,14) which are capable of changing the valence states of U(IV) or U(VI) (15-17) and therefore possibly the amount of uranium lost during decladding. The dissolution rate of uranium dioxide in sulfuric acid was found to be directly proportional to oxygen overpressure (18).

Laboratory work was performed by G. E. Woodall, D. M. Helton, and E. R. Johns. Analyses were made by G. R. Wilson's group of the Analytical Chemistry Division. The irradiated Consolidated Edison pellet used in Sect. 3.0 was supplied by J. H. Kittel, Metallurgy Division, Argonne National Laboratory.

2.0 DECLADDING LOSSES

A large increase in uranium losses, previously observed (9) when air was introduced during Sulfex decladding of uranium oxide fuel, was also observed in this work with Consolidated Edison type fuel pellets. Irradiation increased the losses in the final solutions much less than did introduction of air. The results indicated that air should not be admitted to the system during decladding, particularly in Darex decladding, and that refluxing initial Sulfex solution should

not be permitted to contact Consolidated Edison pellets, particularly if air is admitted to the system. Even with air excluded, uranium losses at the boiling point increased almost linearly to 1% in 30 hr in both irradiated initial Sulfex and initial Darex solutions. Final decladding solutions should be removed from the pellets as quickly as practical, but, if cooled, they may stay in the dissolver several hours without serious uranium or thorium losses.

2.1 Losses Caused by Exposure to Air at the Boiling Point

In Final Sulfex and Darex Solutions. Exposure of refluxing final Darex decladding solution to air (no irradiation) while contacting 6% uranium dioxide—94% thorium dioxide pellets caused the total uranium losses to more than triple, from 0.25 to 0.90%, in 3 hr contact time (Fig. 1a and Table 1) and increase about 0.05%/hr thereafter while the incremental uranium loss over the 0.19% at the start of the experiment increased from 0.06 to 0.71% in 3 hr. Similar treatment with final Sulfex solution caused the total uranium losses to increase 30%, from 0.06 to 0.08%, in 1 hr and to increase 0.01% per hour thereafter while the incremental uranium loss over the 0.04% at the start of the experiment increased from 0.02 to 0.04% in 1 hr. A similar but somewhat smaller effect on thorium losses was observed in refluxing final Darex solution, the thorium losses being almost immeasurably small in refluxing final Sulfex solution (Fig. 1b and Table 1). Figure 1 shows that exclusion of air is particularly important in Darex decladding.

In Initial Sulfex and Darex Solutions. In practice, the average composition of the decladding solution contacting the core will lie between the initial and final solutions, and it was necessary to determine if large uranium and thorium losses occur during contact with the initial decladding solutions. Losses for the first 20 hr with air excluded were smaller in the initial Sulfex and Darex (Figs. 2a and b) solutions than in the final solutions and increased rapidly when the initial solutions were exposed to air.

In refluxing initial Sulfex solution not exposed to air, uranium losses were only 0.017% in 3 hr and increased less than 0.01% per hour thereafter (Fig. 2a and Table 1). Exposure to air increased the uranium loss to 0.3% in 3 hr. The rate of increase after 3 hr was only 0.01% per hour. Air apparently caused 3-fold larger uranium losses for the first 3 hr in the initial than in the final Sulfex solution. The soluble thorium losses in initial Sulfex solution were limited to 0.2% by formation of insoluble thorium sulfate (Fig. 2b and Table 1). However, exposure of the refluxing solution to air decreased the time needed to reach the maximum thorium concentration in solution from 20 to 3 hr. This rapid increase in losses when the initial Sulfex solution (exposed to air) first contacts the pellets could cause large losses in a batch Sulfex process in which a large heel of undissolved pellet cores is allowed to accumulate.

In refluxing initial Darex solution, uranium losses increased at a rate of approximately 0.01% per hour and increased further to about 0.02% per hour when the solu-

Table 1. Effect of Exposure to Air and Irradiation on Uranium and Thorium Losses to Initial and Final Decladding Solutions at Boiling Point (see Figs. 1-4)

| Exposure Time, hr | Losses to Initial Decladding Solution, % | | | | | | Losses to Final Decladding Solution, % | | | | | |
|-------------------|--|-------|---------|------|------------|------|--|-------|---------|-------|------------|-------|
| | Untreated | | Aerated | | Irradiated | | Untreated | | Aerated | | Irradiated | |
| | U | Th | U | Th | U | Th | U | Th | U | Th | U | Th |
| Darex | | | | | | | | | | | | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0.19 | 0.10 | 0.19 | 0.10 | 0.19 | 0.10 |
| 1 | 0.04 | a | 0.02 | a | 0.02 | a | b | b | 0.65 | b | b | b |
| 3 | 0.07 | 0.02 | 0.06 | 0.04 | 0.08 | 0.08 | 0.25 | 0.14 | 0.90 | 0.20 | b | 0.15 |
| 10 | 0.15 | 0.05 | 0.19 | 0.15 | 0.30 | 0.30 | 0.34 | 0.16 | 1.25 | 0.42 | 0.27 | 0.18 |
| 25 | 0.30 | 0.15 | 0.50 | 0.40 | 0.88 | 0.85 | 0.38 | 0.20 | 1.97 | 0.91 | 0.35 | 0.26 |
| 30 | 0.34 | 0.2 | 0.60 | 0.50 | 1.10 | 1.04 | 0.40 | 0.21 | 2.10 | 1.06 | 0.38 | 0.29 |
| 70 | - | - | - | - | - | - | 0.55 | 0.32 | - | - | 0.60 | 0.50 |
| Sulfex | | | | | | | | | | | | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0.04 | 0.005 | 0.04 | 0.005 | 0.04 | 0.005 |
| 1 | 0.010 | a | 0.19 | 0.08 | 0.025 | a | 0.06 | b | 0.08 | b | 0.069 | b |
| 3 | 0.017 | 0.025 | 0.29 | 0.19 | 0.086 | 0.03 | 0.064 | b | 0.09 | b | 0.086 | b |
| 10 | 0.05 | 0.07 | 0.41 | 0.19 | 0.30 | 0.11 | 0.078 | b | 0.15 | b | 0.118 | b |
| 25 | 0.14 | 0.15 | 0.57 | 0.19 | 0.75 | 0.22 | 0.106 | 0.04 | 0.26 | 0.055 | 0.161 | 0.03 |
| 30 | 0.18 | 0.15 | 0.62 | - | 0.89 | 0.22 | 0.115 | 0.05 | 0.295 | 0.067 | 0.176 | 0.04 |

^aBelow measurable concentration.

^bNo measurable change.

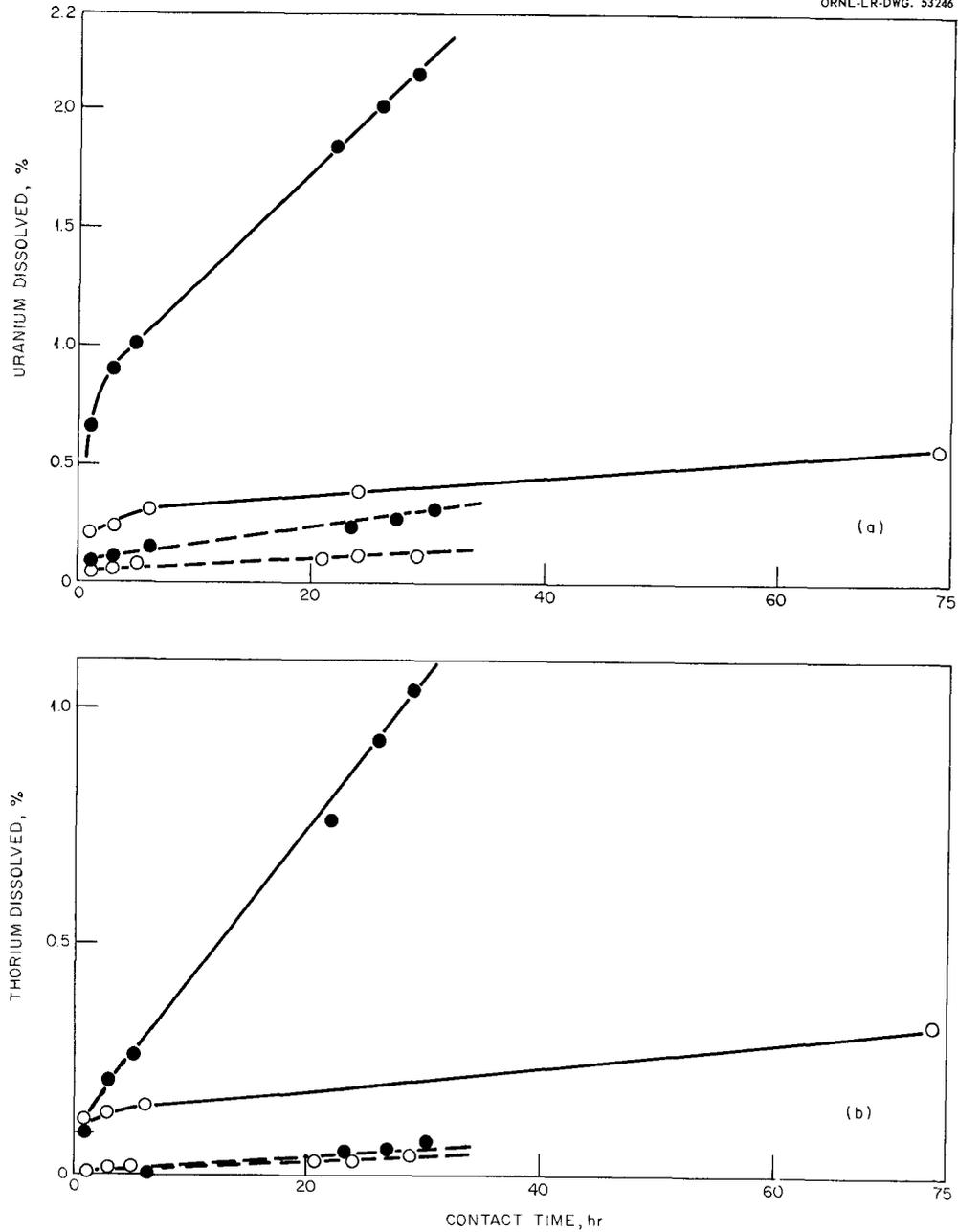


Fig. 1. (a) Uranium and (b) thorium losses from Consolidated Edison type fuel pellets to final Darex (—) and Sulfex (---) solutions at the boiling point. ○ untreated; ● exposed to air. Solution concentrations at start: Darex, 0.012 mg U/ml, 0.18 mg Th/ml, 0.084 M SS, 2.9 M H⁺, these uranium and thorium values being equivalent to 0.19 and 0.1% losses to decladding solution, respectively; Sulfex, 0.003 mg U/ml, 0.013 mg Th/ml, 1 M SS, 5.7 M H⁺, these uranium and thorium values being equivalent to 0.04 and 0.005% losses to decladding solution, respectively. Pellets: ~6% uranium dioxide, 94% thorium oxide, O/U = 2.4, 93% of theoretical density.

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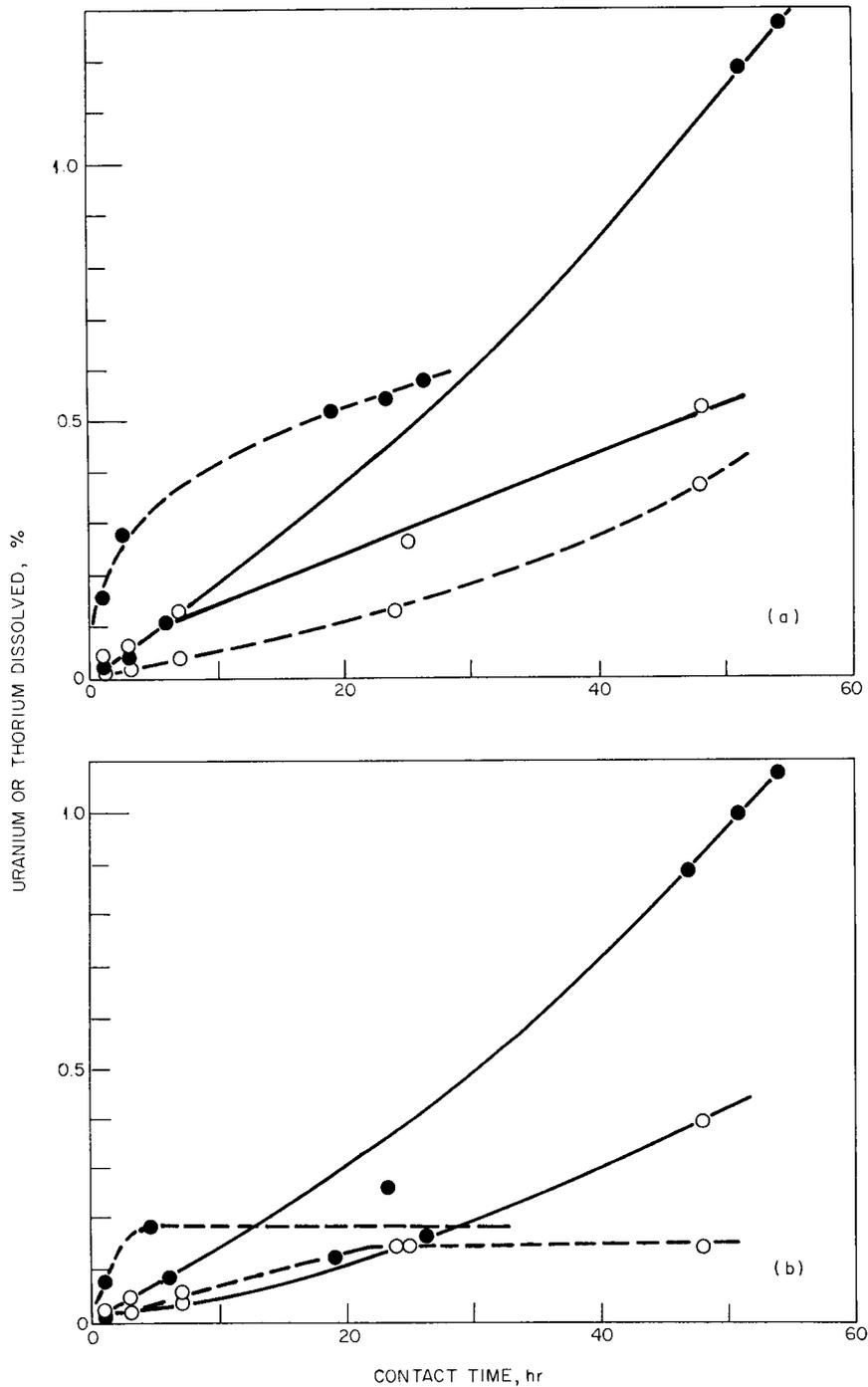


Fig. 2. (a) Uranium and (b) thorium losses from Consolidated Edison type fuel pellets to initial Darex (—) and Sulfex (---) solutions at the boiling point. ○ untreated; ● exposed to air. Solution concentrations at start: Darex: 5 M HNO₃-2 M HCl; Sulfex: 6 M sulfuric acid; pellets: ~6% uranium dioxide, 94% thorium dioxide, O/U = 2.4, 93% of theoretical density.

tion was exposed to air (Fig. 2a and Table 1). Thorium losses followed uranium losses closely, but were slightly smaller (Fig. 2b and Table 1).

2.2 Losses Caused by Irradiation at the Boiling Point

Irradiation with Co-60 radiation at a radiant power density of about 1 watt/liter, which is approximately the density expected in actual processing (19), produced no significant change in the uranium losses to refluxing final Darex solution over 70 hr (Fig. 3a and Table 1). Thorium losses for the first 3 hr were not increased by the irradiation, but appeared to increase slightly after 3 hr. In the refluxing final Sulfex solution, similar irradiation appeared to cause about a 30% increase, from 0.064 to 0.086%, in uranium losses in 3 hr and had little additional effect thereafter (Fig. 3b and Table 1). No significant change in thorium losses was observed during irradiation of final Sulfex solution.

Irradiation of refluxing initial Sulfex (Fig. 4a) and Darex (Fig. 4b) solutions caused up to 5- and 3-fold increases in uranium losses, respectively, throughout dissolution. Thorium losses were smaller than uranium, and the maximum thorium loss was 0.2% in initial Sulfex solutions, due to formation of insoluble thorium sulfate. Fortunately, actual losses in processing should be closer to those observed in final Sulfex and Darex solutions (Fig. 3), where irradiation produced only a minor change in losses. It is interesting to note that uranium losses were greater in irradiated (Fig. 4b) than in aerated (Fig. 2a) initial Darex solution. In irradiated initial Sulfex solution (Fig. 4a), uranium losses exceed those in aerated solution (Fig. 2a) after 16 hr contact time. A large buildup of undissolved core particles through several cycles apparently could lead to appreciable uranium loss during decladding in a batch process when the fresh decladding solution is introduced.

2.3 Losses at 33°C

At 33°C, uranium and thorium losses to the final Sulfex solutions were approximately equal to those at the boiling point at the end of the first hour (Fig. 3b), but increased only 0.0004% per hour thereafter. Irradiation had no measurable effect. A very small uranium loss of 0.006% had occurred after 5 hr in initial Sulfex solution without irradiation, but irradiation doubled the uranium loss and increased the rate of uranium loss thereafter from less than 0.0001 to 0.0002% per hour (Fig. 5). Uranium and thorium losses to initial Darex decladding solution at 33°C, both irradiated and nonirradiated, did not change during 5 hr contact. These results indicate that losses from high-density Consolidated Edison pellets will not increase significantly if the decladding solution should be left in the dissolver in contact with the pellets for several hours, provided that the solution is cooled to ambient temperature.

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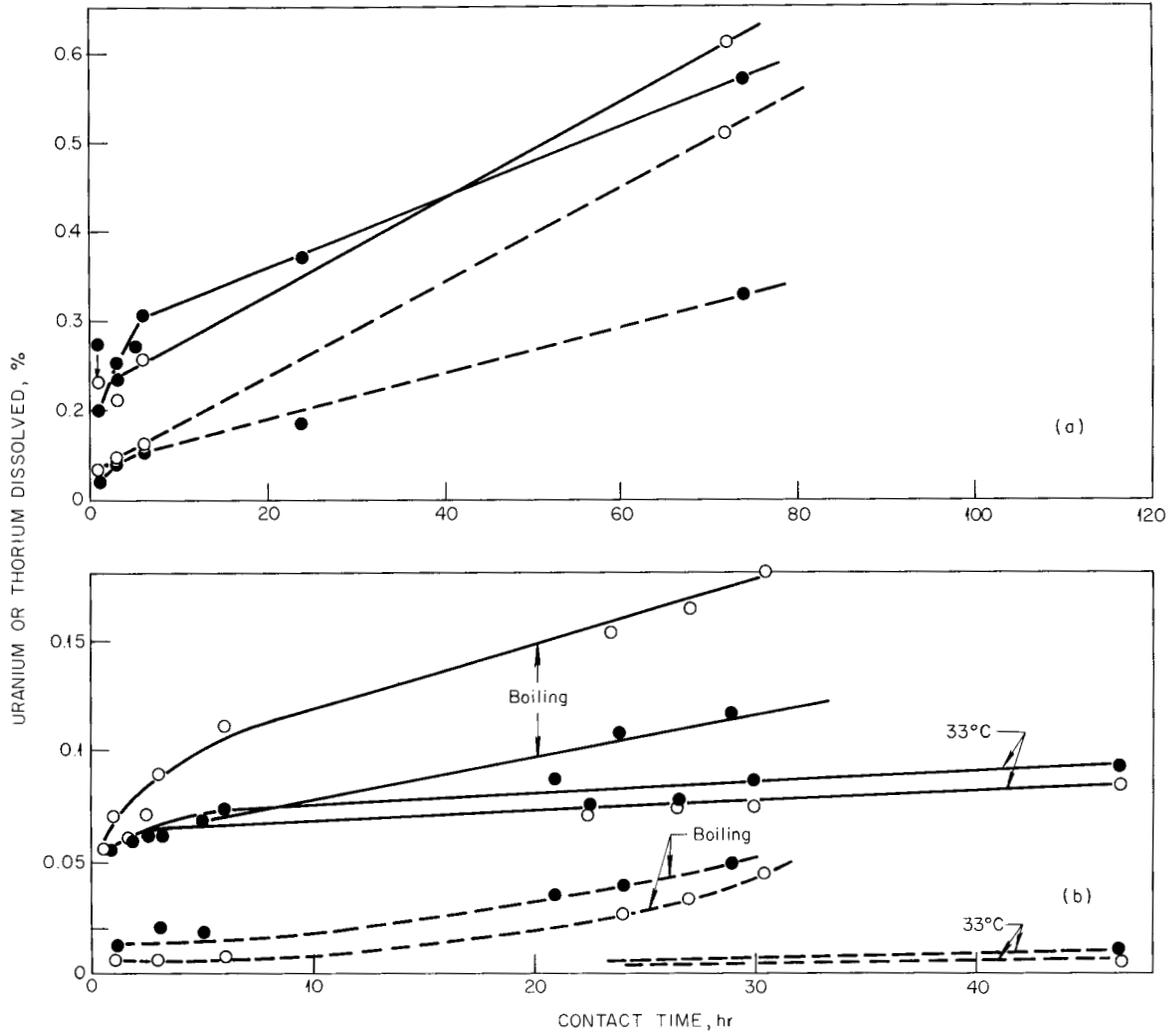


Fig. 3. Uranium (—) and thorium (---) losses from Consolidated Edison type pellets to final (a) boiling Darex and (b) Sulfex solutions. Solution concentrations at start: see Fig. 1. Pellets: O/U = 2.4, 93% of theoretical density. Radiant power density: 1 watt/liter. O irradiated; ● control.

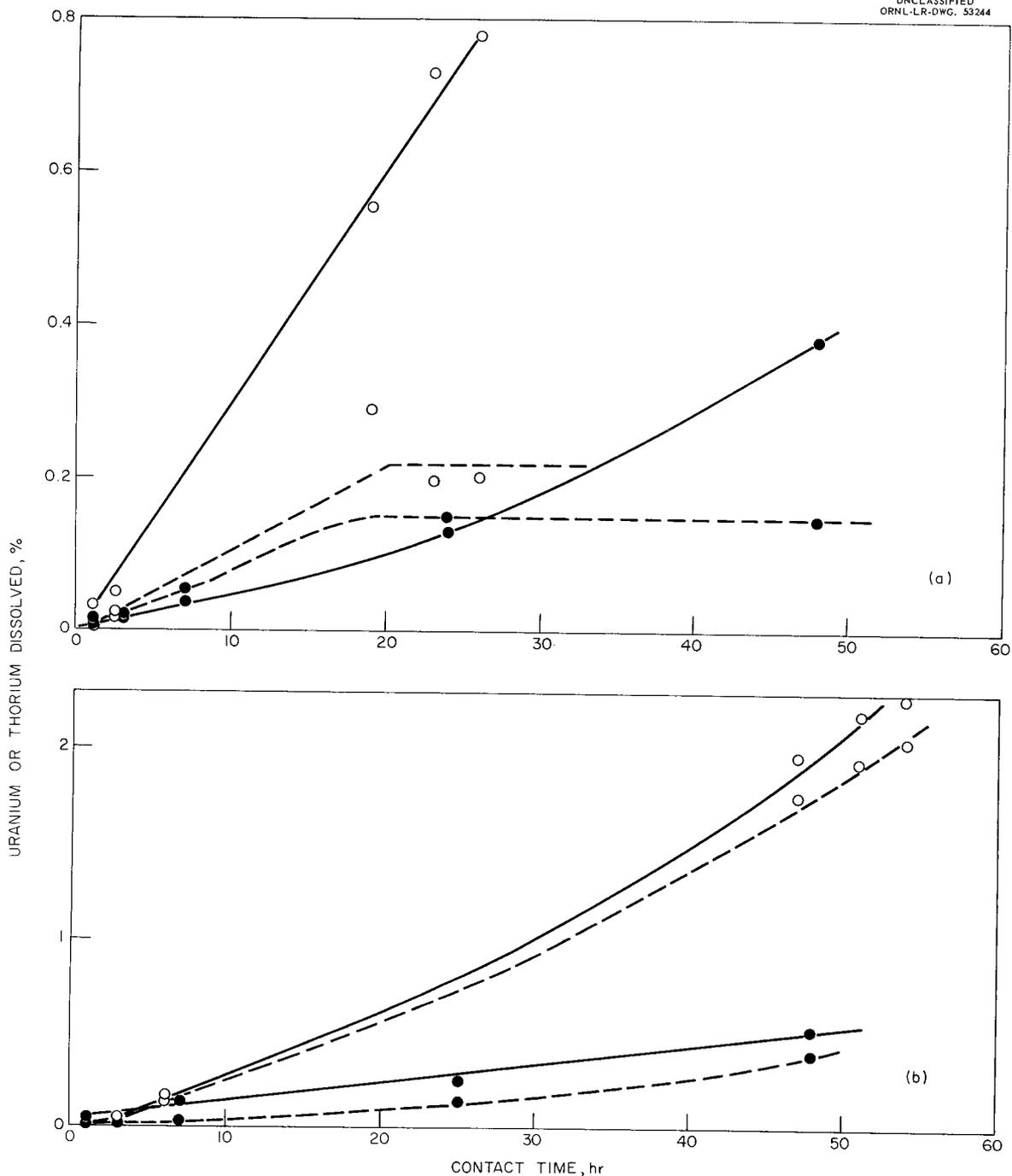


Fig. 4. Uranium (—) and thorium (---) losses from Consolidated Edison pellets to initial (a) Sulfex and (b) Darex solution at the boiling point. Solution concentrations at start: see Fig. 2. Pellet: O/U = 2.4, 93% of theoretical density. Radiant power density: 1 watt/liter. O irradiated; ● control.

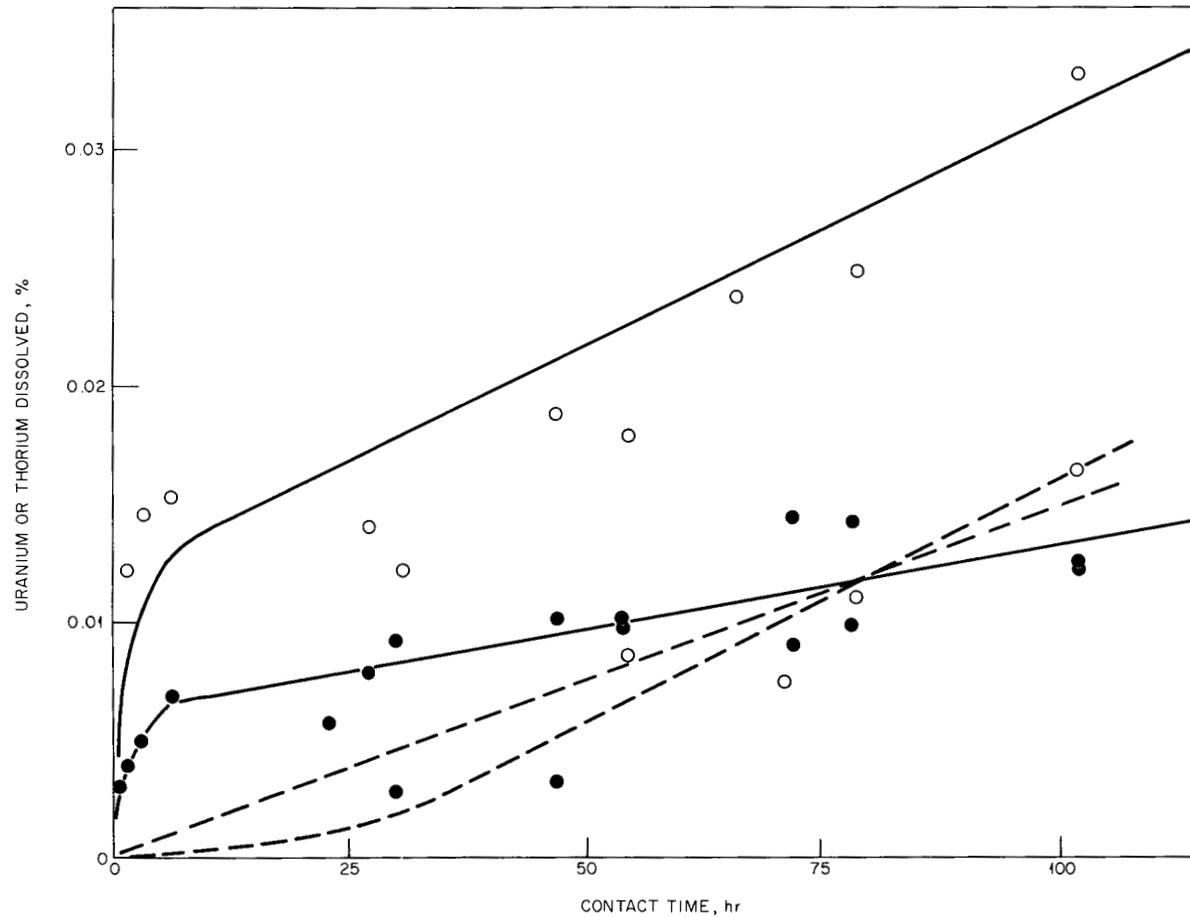


Fig. 5. Uranium (—) and thorium (---) losses from a Consolidated Edison type pellet to initial Sulfex solution at 33°C. Solution concentration at start: 6 M sulfuric acid. Pellet: O/U = 2.4, 93% of theoretical density. Radiant power density: 1 watt/liter. O irradiated; ● control.

2.4 Material Used

Consolidated Edison type fuel pellets obtained from the Davison Company (prepared by treating air-fired pellets with hydrogen at 600°C, in which the uranium was present as UO₂, 93% of theoretical density), were immersed for periods of time of up to 70 hr in flowsheet volumes of initial or final Sulfex or Darex decladding reagents. Aliquots of the reagent (1 ml) were periodically removed and analyzed for uranium and thorium. The solution removed was replaced with fresh reagent. Samples to be irradiated were placed in a thermostatted vessel in the 1000-curie Co-60 source, which produced a radiant power density of about 1 watt/liter, approximately that expected in actual processing (19). Exposure to air was effected simply by removing the plastic tube and water trap from the exit of the short (3-in.) condenser.

Initial Sulfex and Darex solutions are 6 M sulfuric acid and 5 M nitric acid—2 M hydrochloric acid, respectively. The final decladding solutions containing dissolved stainless steel were diluted slightly to ensure stability at the 33°C temperature used in some experiments (Sect. 2.3). The slightly diluted final Sulfex solution contained 5.7 N H⁺, 0.71 M Fe(II), 0.1 M Ni, 0.19 M Cr, 0.003 mg/ml U, 0.013 mg/ml Th, and the Darex solution contained 2.9 N H⁺, 0.6 M Fe(III), 0.1 M Ni, 0.14 M Cr, 0.012 mg/ml U, and 0.18 mg/ml Th before use in these runs.

To aid in intercomparison of data from many runs, loss values at certain time intervals were taken from the smooth curves drawn through the Sulfex and Darex experimental points (Table 1).

3:0 LOSSES FROM A CRUSHED, IRRADIATED CONSOLIDATED EDISON PELLET TO INITIAL SULFEX SOLUTION

A single experiment was performed with a crushed neutron-irradiated (5600 Mwd/ton) Consolidated Edison type pellet (97.5% ThO₂—UO₂), decayed 3.8 years, to investigate uranium and thorium losses that might be expected under highly unfavorable conditions during Sulfex decladding. Refluxing initial Sulfex solution, in which losses increased more rapidly than in the final Sulfex solution (Table 1), was used. The top of the condenser was open to the atmosphere. The pellet, which had been clad in 304 stainless steel (air bonded) in the reactor, was exposed to the air for about 1 week after mechanical decladding and crushing. The surface area of the oxide particles was not measured.

Uranium losses were nearly 3% after 3 hr contact time and about 15% after 50 hr (Fig. 6). Because of the low radiation power density (estimated at 10⁻³ watt/liter), the high uranium losses must be ascribed to surface dissolution reactions and not to irradiation. This result indicates that high losses might occur during decladding of Consolidated Edison pellets if the pellets become shattered during neutron-irradiation, transfer, or shipment and are permitted to contact the initial aerated Sulfex solution.

However, the density of the pellet used in the above experiment was unknown and was probably lower than that of pellets that will be used in the Consolidated Edison Reactor (93% of theoretical). The denser pellets have been shown to produce lower losses (7).

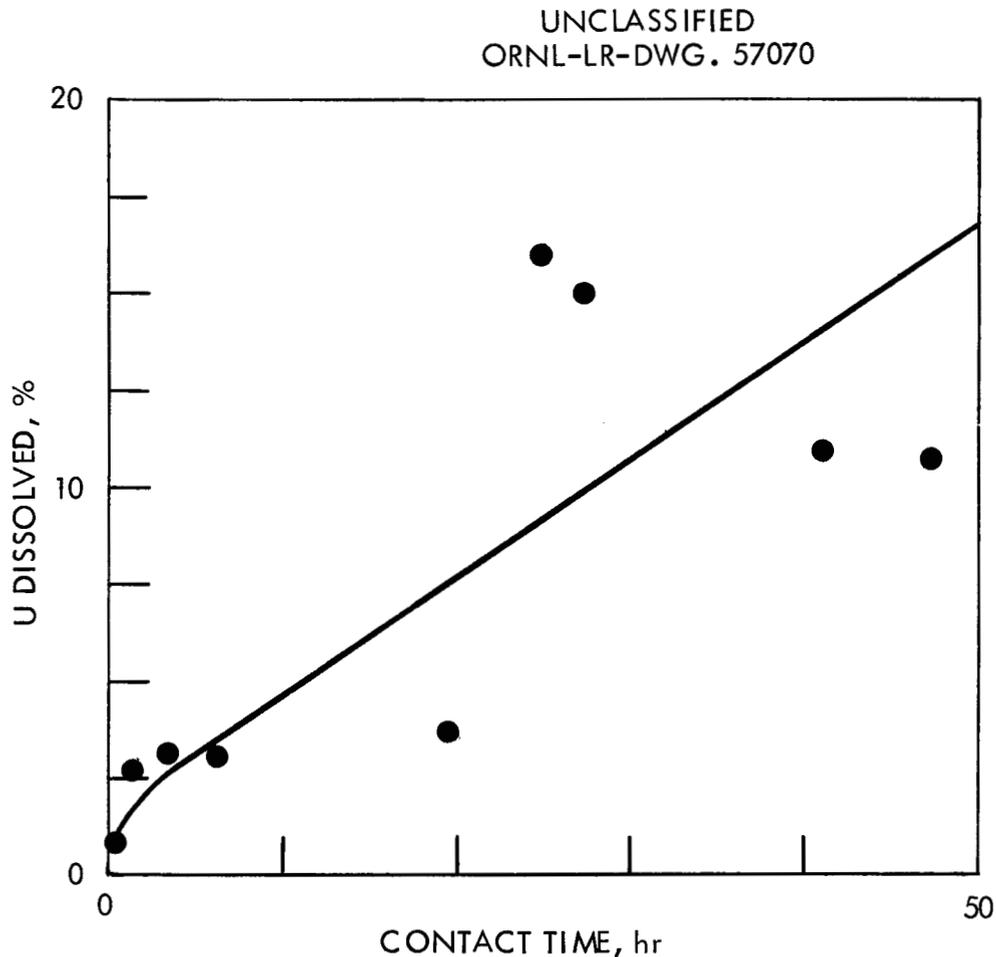


Fig. 6. Uranium losses from a crushed irradiated Consolidated Edison type pellet to refluxing 6 M sulfuric acid. Burnup 5600 Mwd/ton; decayed 3.8 years; radiant power density $\sim 10^{-3}$ watt/liter; pellet weight 2.7 g (2.5% UO_2); 11.5 cc of 6 M H_2SO_4 .

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