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MINIMUM CRITICAL ^{235}U ENRICHMENT OF HOMOGENEOUS HYDROGENOUS URANYL NITRATE

S. R. Bierman and G. M. Hess

Pacific Northwest Laboratory

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CRITICALITY DATA CENTER

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JUNE 1968

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FOREWORD

The Criticality Data Center has been established at the Oak Ridge National Laboratory under the auspices of the U. S. Atomic Energy Commission for the development of methods allowing extension and application of data derived from experiments and from analyses to problems in nuclear criticality safety, as well as for the review and evaluation of the data themselves. A necessary part of this program is a medium whereby information germane to the intent of the Center is made available. This report series has been inaugurated for that purpose. Inquiries should be directed to E. B. Johnson, P. O. Box Y, Oak Ridge, Tennessee 37830.

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MINIMUM CRITICAL ^{235}U ENRICHMENT OF HOMOGENEOUS
HYDROGENOUS URANYL NITRATE

S. R. Bierman and G. M. Hess

ABSTRACT

A series of experiments with 2.14 and 2.26 wt % ^{235}U -enriched uranyl nitrate was performed in the Physical Constants Test Reactor (PCTR) at the Pacific Northwest Laboratory to determine the minimum critical ^{235}U enrichment for homogeneous hydrogenous uranyl nitrate and to observe the reactivity effect of neutron moderation on these low enriched uranium materials. The minimum critical enrichment was determined to be 2.104 ± 0.010 wt % ^{235}U at an optimum H:U atomic ratio of 7.6. Optimum neutron moderation occurred, with k_{∞} values of 1.013 and 1.04, at H:U atomic ratios of 8.0 ± 1.0 and 9.3 ± 0.5 , respectively, for the 2.14 and 2.26 wt % enriched uranyl nitrate. Values of k_{∞} greater than unity were observed for H:U ratios between 6 and 10.5 for the 2.14 wt % enrichment and between 4 and 15 for the 2.26 wt % enrichment.

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INTRODUCTION

In the recovery of uranium from spent fuel elements and uranium-bearing wastes, the uranium is converted to the nitrate form by dissolution in a nitric acid solution. It remains in this chemical form throughout many of the processing and handling steps associated with its recovery. Consequently, a firm knowledge is needed of the minimum ^{235}U enrichment for which a homogeneous uranyl nitrate system can be made critical. This minimum critical enrichment is the enrichment required to obtain an infinite neutron multiplication factor, k_{∞} , of unity under conditions of optimum moderation. Below this enrichment, criticality would not be possible during the processing and handling of the uranyl nitrate. Also, this enrichment and the H:U ratio of the homogeneous mixture that gives a maximum k_{∞} of unity provide a fundamental point for comparison with reactor theory. It is also desirable, when checking calculational models and establishing nuclear safety guidelines, to know over what range of moderation criticality is possible for a given enrichment. This is particularly true for low enriched uranium systems, since at low enrichments criticality is strongly dependent on both neutron moderation and ^{235}U enrichment.

An experimental program to provide the above information was completed in the Physical Constants Test Reactor (PCTR) of the Pacific Northwest Laboratory.* The results and interpretation of these measurements and correlated data from some previous PCTR measurements are presented herein. The previous experiments¹ performed in the PCTR showed the minimum critical ^{235}U enrichment for homogeneous mixtures of UO_3 in water to be 1.034 ± 0.010 wt %. Additional experiments² with 3.04 wt % enriched UO_3 and uranyl nitrate, $\text{UO}_2(\text{NO}_3)_2$, homogeneously mixed with hydrogenous substances established that the presence of the nitrate radical had a pronounced effect on criticality. The 3.04 wt % enriched UO_3 had a maximum

*A summary of this work appeared in Nucl. Sci. Eng. 32, 135 (April 1968).

1. V. I. Neeley and H. E. Handler, "Measurement of Multiplication Constant for Slightly Enriched Homogeneous UO_3 -Water Mixtures and Minimum Enrichment for Criticality," HW-70310, Hanford Atomic Products Operation (1961).
2. V. I. Neeley, J. A. Berberet, and R. H. Masterson, " k_{∞} of Three Weight Per Cent UO_3 and $\text{UO}_2(\text{NO}_3)_2$ Hydrogenous Systems," HW-66882, Hanford Atomic Products Operation (1961).

k_{∞} value of 1.350 ± 0.013 as compared to 1.145 ± 0.01 for the 3.04 wt % uranyl nitrate. Subsequent calculations^{3,4} indicated that the minimum critical enrichment for homogeneous hydrogenous uranyl nitrate systems was about 2 wt %. Consequently, experiments were planned for ^{235}U enrichments slightly above this value.

3. K. R. Ridgway, Trans. Am. Nucl. Soc. 9, 134 (1966).

4. K. R. Ridgway, "Calculated Critical Parameters of Low Enrichment UNH and $\text{UO}_3\text{-H}_2\text{O}$ Mixtures," ISO-174, Isochem, Inc. (1966).

THEORETICAL BASIS

The theory on which the PCTR technique is based has been adequately covered in other publications^{1,2,5,6}. Only a short summary will be presented in this report to establish the basic relationship needed for analyzing the experimental data.

The basic principle involved in making reactivity measurements in the PCTR is that if any volume of an infinite homogeneous critical medium is replaced with a vacuum, there would not be any change in k_{∞} from unity, since there would be no change in the neutron density or in the energy distribution. Infinite systems are simulated in the PCTR to determine at what compositions this phenomenon occurs and to determine how far k_{∞} is from unity for different material compositions.

The stationary part of the PCTR is shown in Fig. 1. The control and safety rods are contained in this portion of the PCTR in addition to the driver fuel elements and the central cavity in which the test material is positioned. The neutron flux that is established in the driver region is characteristic of ^{235}U in graphite. Sufficient test material must be placed in the central cavity to modify this ^{235}U -graphite spectrum to one that is characteristic of only the test material over at least a center portion of this material. The neutron multiplication in this center region is then a function only of the material in this region and is independent of neutron leakage.

If a volume of this center region, where the neutron flux is said to be matched, is replaced with a void, it is possible to determine if the infinite neutron multiplication factor, k_{∞} , is equal to, less than, or greater than unity. If the reactivity response of the PCTR to the void is ρ_v and to the material ρ_o , then

$$\text{if } \rho_o < \rho_v, k_{\infty} < 1,$$

$$\text{if } \rho_o = \rho_v, k_{\infty} = 1,$$

$$\text{if } \rho_o > \rho_v, k_{\infty} > 1.$$

5. D. J. Donahue *et al.*, Nucl. Sci. Eng. 4, 297 (1958).

6. R. E. Heineman, "Experience in the Use of the Physical Constants Testing Reactor," Peaceful Uses of Atomic Energy, 12, 650 (1958).

Fig. 1. Typical PCTR Loading with 21-in. Cube of Uranyl Nitrate in Center Cavity.

At only two concentrations of fuel-moderator will a homogeneous mixture have a k_{∞} of unity. How k_{∞} of other concentrations of this material differ from unity can be determined by observing the reactivity response, ρ_1 , of the PCTR when a neutron absorbing material is added to or removed from the mixture. By knowing exactly how much neutron absorbing material is required for ρ_1 to equal ρ_v , it is possible to determine k_{∞} for the material in the absence of absorber.

The infinite neutron multiplication factor for the fuel-moderator can be calculated by

$$k_{\infty} = \eta f \epsilon p \quad (1)$$

Since

$$\eta f = \left[\frac{\nu \Sigma_f \phi_v}{\Sigma_a \phi_v} \right]_{\text{FUEL}} \left[\frac{\Sigma_a^{\text{FUEL}}}{\Sigma_a^{\text{MOD}} + \Sigma_a^{\text{FUEL}}} \right],$$

$$k_{\infty} = \frac{\left[\epsilon p \nu \Sigma_f \phi_v \right]_{\text{FUEL}}}{\left[\Sigma_a^{\text{MOD}} + \Sigma_a^{\text{FUEL}} \right] \phi_v} \quad (2)$$

Similarly, k_{∞}' for the material containing a neutron absorber can be calculated by

$$k_{\infty}' = \frac{\left[\epsilon p \nu \Sigma_f \phi_v \right]_{\text{FUEL}}'}{\left[\Sigma_a^{\text{ABS}} + \Sigma_a^{\text{MOD}} + \Sigma_a^{\text{FUEL}} \right]' (\phi_v)'} \quad (3)$$

If this mixture contains exactly the right amount of neutron absorbing material for $\rho_v = \rho_1$, k_{∞}' is unity. At this condition the ratio of Eq. (2) to (3) yields an expression for k_{∞} of the fuel-moderator

$$k_{\infty} = \frac{\epsilon p \left[\nu \Sigma_f \phi_v \right]_{\text{FUEL}}}{\left[\Sigma_a^{\text{MOD}} + \Sigma_a^{\text{FUEL}} \right] \phi_v} \cdot \frac{\left[\Sigma_a^{\text{ABS}} + \Sigma_a^{\text{MOD}} + \Sigma_a^{\text{FUEL}} \right]' \phi_v'}{\epsilon' p' \left[\nu \Sigma_f \phi_v \right]_{\text{FUEL}}'} \quad (4)$$

In a homogeneous system the flux terms in Eq. (4) cancel.

$$k_{\infty} = \frac{\epsilon p}{\epsilon' p'} \cdot \frac{\nu \Sigma_f}{\nu' \Sigma_f'} \cdot \frac{[\Sigma_a^{ABS} + \Sigma_a^{MOD} + \Sigma_a^{FUEL}]}{\Sigma_a^{MOD} + \Sigma_a^{FUEL}} \quad (5)$$

Knowing the composition of the material for which k_{∞} is desired and the amount of neutron absorbing material required to change k_{∞} of this material to unity, values for the terms in Eq. (5) can be calculated to obtain k_{∞} for the material. Since Eq. (5) is concerned with ratios only, errors in cross sections are minimized.

EXPERIMENTAL MEASUREMENTS

Measurements were made on uranyl nitrate mixtures having ^{235}U enrichments of 2.14 ± 0.005 wt % and 2.26 ± 0.005 wt % and H:U atomic ratios over the range of about 3 to 12. In this region of moderation uranyl nitrate is in the crystalline form. Consequently, 1/8-in.-diam spheres of linear polyethylene^a having a density of 0.916 g/cm^3 and an H:C atomic ratio of 2 were used to vary the hydrogen content of the sample. Previous measurements² had established that polyethylene was an acceptable replacement for water as a moderator in these systems.

The preparation of the uranyl nitrate, the blending, the loading and unloading of the experimental vessels, and the sampling of the material used in these experiments was carried out by the Chemical Development Section of the Chemistry Department of the Pacific Northwest Laboratory. A detailed description of the material preparation and analysis is contained in Ref. 7. The isotopic composition of the uranium is given in Table A-I of the Appendix to this report.

Since the stable condition for uranyl nitrate is the hexahydrate, it was necessary to remove some of the water of hydration to achieve H:U atomic ratios of less than 12. This was accomplished by temperature control during the crystallization step to obtain a dihydrate. An end product having on the average 1.72 molecules of water per molecule of uranyl nitrate was achieved. This relatively anhydrous salt is deliquescent (see Fig. A-I of the Appendix). Consequently, analyses were made periodically during the course of the experiments to determine the amount of water being picked up by the uranyl nitrate. Based on the degree of hydration at the time of blending, sufficient polyethylene was added to each mixture to achieve the desired H:U ratio.

For making the measurements in the PCTR, each mixture was contained in a 21-in. cube composed of essentially nine 7 x 7 x 21 in. aluminum cans.

^a Eastman Kodak "Tenite"

7. M. R. Schwab, "Informal Critical Safety Analysis Report for UND Project, Parts I and II," BNWL-CC-1427-PT 1, Pacific Northwest Laboratory (1967).

The center 7 x 7 x 21-in. can actually consisted of three 7-in. cubes, the center one being the aforementioned center region, or test cell, over which changes in neutron multiplication were observed. A typical loading of the PCTR for these experiments is shown in Fig. 1. A better perspective of the two types of cans making up the 21-in. cube can be obtained from Fig. 2.

The usual criterion of a constant cadmium ratio⁶ was used to establish that a characteristic flux existed across the test material in each experiment. Using gold foils, cadmium ratios were determined in both the radial and the axial directions across the entire 21-in. cube and on the face of the PCTR graphite opposite these traverses. The foil traverse rods and their relative positions in the material are shown in Fig. 2.

The reactivity response of the PCTR to 13 different fuel-moderator mixtures was observed to establish the reactivity worth of each mixture relative to k_{∞} of unity. Each of these measurements was made by observing the excess reactivity of the PCTR under a certain set of conditions with the center test cell void and then observing the change in reactivity when this void was filled with the same, or similar, material as the rest of the 21-in. cube. The results of these measurements are shown in Fig. 3 as a function of the H:U ratio and ²³⁵U enrichment. The curves are least-square fits.

With the high density (2.75 g/cm³ as compared to 2.802 g/cm³ theoretical density for UNH) obtained in the preparation of the uranyl nitrate, it was possible to make accurate reactivity measurements on test cells having H:U ratios differing from the rest of the material by as much as 1.5 units. Reactivity measurements thus made were within the reproducibility of the measurements made when the center test cell and the rest of the mixture were identical in composition. The results of these measurements are included in Table A-II of the Appendix.

Except as noted in Fig. 3, the material in the test cell and the rest of the 21-in. cube was the same for each of the 2.14 wt % enriched mixtures. To help establish an upper bound on the 2.14 wt % enrichment curve shown in Fig. 3, measurements were also made in the 7.24 H:U mixture with test cells having H:U atomic ratios of 8.08 and 9.25. Since both

Fig. 2. Uranyl Nitrate Containers and Foil Traverse Holders.

Fig. 3. Measured Reactivity Response of the PCTR to Uranyl Nitrate as a Function of Neutron Moderation.

the 8.08 and 9.25 H:U test cells are rich in hydrogen relative to the undermoderated 7.24 H:U spectrum, the reactivity response of the PCTR to these two test cells would be expected to be greater than if each test cell were in a more characteristic neutron spectrum. Thus, the reactivity curve, as a function of H:U, should lie somewhere below the observed values for these two test cells.

For the 2.26 wt % enrichment measurements, two different mixtures having H:U atomic ratios of 11.2 and 8.25 were used with test cells having H:U ratios as indicated in Fig. 3 and in Table A-II of the Appendix.

RESULTS AND INTERPRETATIONS

Based on least-squares fitting of the results shown in Fig. 3, optimum neutron moderation for 2.14 wt % ^{235}U enriched uranyl nitrate occurs at an H:U atomic ratio of 8.0 ± 1.0 and for 2.26 wt % ^{235}U enriched uranyl nitrate at 9.3 ± 0.5 . These values are in line with the optimum H:U of about 10.5 observed in the 3.04 wt % enriched uranyl nitrate experiments.³

As can be seen in Fig. 3, the 2.14 wt % enriched material has an excess reactivity of only 1.2 cents above a k_{∞} of unity at optimum neutron moderation and thus is very near the minimum critical enrichment. A linear extrapolation of the maximum excess reactivity as a function of ^{235}U enrichment, shown in Fig. 4, yields a minimum critical enrichment of 2.104 ± 0.01 wt %. When treated in the same manner, calculations by Ridgway,⁴ using the GAMTEC-II Computer Code,⁸ were found to agree quite well with this value, as is indicated in the figure.

In addition to the reactivity response of the PCTR to each of the mixtures shown in Fig. 3, reactivity measurements were also made on each material shown in Table 1 to determine the amount of neutron absorbing material, in the form of borated polyethylene, required to achieve a null reactivity between the test cell void and the test cell filled with the mixture of fissile, moderating, and absorbing materials. At least two boron concentrations were investigated. From a least-squares fit of these data, the amount of boron required for the null reactivity condition can be predicted, as shown in Fig. 5, for a typical set of measurements. The results and the corresponding boron concentrations are tabulated in Table A-II of the Appendix for each material. The B: ^{235}U atomic ratio required to achieve a null reactivity condition for each material is shown in Table 1. Values of k_{∞} for each material were calculated from these data, Eq. (5), and the necessary cross sections obtained from GAMTEC-II code.⁸ The values of k_{∞} were then corrected for the reactivity effect of small amounts of copper, zirconium, and iron contaminants, present in each mixture, by the

8. L. L. Carter, C. R. Richey, and C. E. Hughey, "GAMTEC-II: A Code for Generating Consistent Multigroup Constants Utilized in Diffusion and Transport Theory Calculations," BNWL-35, Pacific Northwest Laboratory (1965).

Fig. 4. Maximum PCTR Excess Reactivity as a Function of ^{235}U Enrichment.

Table 1. Compilation of Data - PCTR Uranyl Nitrate Experiments

Experiment	6702-2	6702-3	6702-4	6702-5	6702-6	6702-7	6702-8	6702-9	6702-10	6702-11	6702-12
Composition (atoms/b cm)											
²³⁸ U	0.37096E-2 ^a	0.32298E-2	0.32298E-2	0.26522E-2	0.26522E-2	0.26522E-2	0.29610E-2	0.29610E-2	0.30261E-2	0.29571E-2	0.26470E-2
²³⁵ U	0.81898E-4	0.71306E-4	0.71306E-4	0.58888E-4	0.58888E-4	0.58888E-4	0.65348E-4	0.65348E-4	0.66982E-4	0.69419E-4	0.62063E-4
N	0.76619E-2	0.66004E-2	0.66004E-2	0.55901E-2	0.55901E-2	0.55901E-2	0.60854E-2	0.60854E-2	0.63749E-2	0.66326E-2	0.59390E-2
O	0.37189E-1	0.32665E-1	0.32665E-1	0.27337E-1	0.27337E-1	0.27337E-1	0.30592E-1	0.30592E-1	0.31259E-1	0.32520E-1	0.29112E-1
H	0.13281E-1	0.21005E-1	0.21005E-1	0.28087E-1	0.28210E-1	0.28210E-1	0.25603E-1	0.25603E-1	0.22177E-1	0.24969E-1	0.30341E-1
C	0.00000	0.42161E-2	0.42161E-2	0.89650E-2	0.89650E-2	0.89650E-2	0.65057E-2	0.65057E-2	0.51203E-2	0.58897E-2	0.92700E-2
Cu	0.43638E-4	0.39243E-4	0.39243E-4	0.58740E-4	0.58740E-4	0.58740E-4	0.35477E-4	0.35477E-4	0.67562E-4	0.49986E-4	0.42507E-4
Zr	0.10728E-3	0.96649E-4	0.96645E-4	0.14508E-3	0.14508E-3	0.14508E-3	0.89581E-4	0.89581E-4	0.16472E-3	0.85462E-4	0.76982E-4
Fe	0.12850E-4	0.87782E-5	0.87782E-5	0.82363E-5	0.82363E-5	0.82363E-5	0.10091E-4	0.10091E-4	0.10249E-4	0.77551E-5	0.72543E-5
²³⁸ U Enrichment (wt %)	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.14 ± 0.005	2.26 ± 0.005	2.26 ± 0.005
H:U ^b	3.49 ± 0.09	6.36 ± 0.14	6.36 ± 0.14	10.36 ± 0.10	10.40 ± 0.17	10.40 ± 0.17	8.46 ± 0.15	8.46 ± 0.15	7.17 ± 0.17	8.25 ± 0.19	11.2 ± 0.19
B: ²³⁸ U @ k _∞ = 1		0.0025 ± 0.0012	0.0024 ± 0.0018	-0.006 ± 0.011	-0.0059 ± 0.011	-0.0055 ± 0.0107	0.0138 ± 0.0009	0.0133 ± 0.0025	0.0060 ± 0.0012	0.0479 ± 0.0004	0.0418 ± 0.0014
Σ _a (cm ⁻¹)		0.05347	0.05347	0.04975	0.04975	0.04975	0.05242	0.05242	0.05189	0.05448	0.05241
Σ _a ^f (cm ⁻¹)		0.05355	0.05355	0.04955	0.04955	0.04955	0.05285	0.05284	0.05207	0.05605	0.05371
ν Σ _f (cm ⁻¹)		0.06875	0.06875	0.06033	0.06033	0.06033	0.06545	0.06545	0.06560	0.06881	0.06376
ν Σ _f ^f (cm ⁻¹)		0.06873	0.06873	0.06036	0.06036	0.06036	0.06536	0.06537	0.06556	0.06847	0.06354
p		0.67470	0.67470	0.74788	0.74788	0.74788	0.71951	0.71951	0.69301	0.71188	0.75538
p ^f		0.67464	0.67464	0.74801	0.74801	0.74801	0.71921	0.71922	0.69287	0.75076	0.75461
k _∞ , Experimental		1.0019 ± 0.0009	1.0017 ± 0.0013	0.9953 ± 0.0086	0.9954 ± 0.0086	0.9957 ± 0.0086	1.0098 ± 0.0003	1.0096 ± 0.0003	1.0045 ± 0.0009	1.0356 ± 0.0003	1.0294 ± 0.0003
Δk ^c											
GAMTEC-II		0.0032	0.0032	0.0048	0.0048	0.0048	0.0031	0.0031	0.0055	0.0032	0.0032
DTP-IV				0.0053	0.0053	0.0053	0.0030	0.0030	0.0053	0.0034	0.0034
k _∞ , Corrected		1.0051 ± 0.0009	1.0049 ± 0.0013	1.0001 ± 0.0086	1.0002 ± 0.0086	1.0005 ± 0.0086	1.0129 ± 0.0003	1.0129 ± 0.0003	1.010 ± 0.0009	1.0393 ± 0.0003	1.0326 ± 0.0003

a. E-2 signifies 10⁻².

b. Determined from uranium and water analyses of uranyl nitrate and the weights of the polyethylene added.

c. Δk_∞ correction for Fe, Cu, and Zr contaminants as calculated by DTP-IV and GAMTEC-II computer codes.

Fig. 5. Typical Excess Reactivity of PCTR as a Function of Boron Concentration in the Homogeneous Uranyl Nitrate Sample.

GAMTEC-II code and the transport theory DTF-IV codes⁹; the corrections given by the two codes were comparable. The results are given in Table 1. The corrected values of k_{∞} are shown graphically in Fig. 6 as a function of H:U together with values of k_{∞} obtained by Neeley² for the 3.04 wt % enriched uranyl nitrate.

The maximum k_{∞} was determined to be 1.0130 ± 0.0003 for the 2.14 wt % enriched uranyl nitrate and about 1.04 for the 2.26 wt % enrichment. Since it is not necessary to establish k_{∞} as a function of composition to determine the minimum critical enrichment of a system, the expense in defining k_{∞} for enrichments only 0.12 percentage points apart was not justified. Consequently, only two experimental determinations of k_{∞} were made for the 2.26 wt % enrichment. The dashed line in Fig. 6 is therefore a best estimate for the 2.26 wt % enriched uranyl nitrate and is based on the other data reported in Fig. 3 as well as that of Fig. 6.

Although the strong effect that neutron moderation and ²³⁵U enrichment have on reactivity can be seen in Fig. 6, the dependence of criticality on both moderation and enrichment as a continuous function can be better seen by crossplotting these data as shown in Fig. 7. The variation in ²³⁵U enrichment with H:U atomic ratio is for a k_{∞} of unity. Therefore, criticality in a hydrogenous, homogeneous uranyl nitrate system is possible only when the composition of the system lies on or above a line through the experimental points of Fig. 7. Below this curve, k_{∞} is less than unity and criticality is not possible even in a system of infinite size. Curves calculated by Ridgway⁴ and by Nichols¹⁰ are also plotted in Fig. 7. That of Ridgway agrees quite well with the experimental data, whereas that of Nichols underestimates the experimental data for the lower enrichments by about 10%, as he predicted it would.

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9. G. B. Carlson et al., DTF Users Manual, United Nuclear Corporation, White Plains, New York (1963).
 10. J. P. Nichols, "Limiting Critical Concentrations of Aqueous Nitrate Solutions of Fissile and Fertile Isotopes," ORNL-TM-686, Oak Ridge National Laboratory (1963).

Fig. 6. k_{∞} as a Function of ^{235}U Enrichment and Neutron Moderation of Infinite Homogeneous Hydrogenous Uranyl Nitrate.

Fig. 7. Critical Concentration of Infinite Homogeneous Uranyl Nitrate as a Function of ^{235}U Enrichment.

The H:U atomic ratio corresponding to the minimum critical ^{235}U enrichment of 2.104 wt % is about 7.6. Since this corresponds to the maximum value of k_{∞} for the 2.104 wt % enriched uranyl nitrate, optimum neutron moderation occurs at an H:U atomic ratio of 7.6.

Another parameter of some use in checking computational models and in criticality control is the amount of neutron absorber required to reduce k_{∞} of a given material to unity. This information is readily available from the measurements with neutron absorbers and is presented in Fig. 8 as a function of the H:U atomic ratio of the 2.14 wt % ^{235}U -enriched homogeneous uranyl nitrate.

Fig. 8. Boron Required in 2.14 wt % ^{235}U Enriched Homogeneous Uranyl Nitrate to Reduce k_{∞} to Unity.

DISCUSSION OF ERRORS

All error limits shown are standard deviations and were determined from propagation of errors in the experimental quantities. These included errors in chemical analyses, material weights, and reactivity measurements. Errors in cross sections were not considered; however, each macroscopic constant was calculated in a neutron spectrum characteristic of the material in question. This approach in conjunction with Eq. (5) tends to minimize errors due to uncertainties in cross sections and it is believed these errors are within the quoted limits.

Errors due to any mismatching of the neutron spectrum were reduced to less than the reproducibility of the reactivity measurements by having an effectively thick buffer region and by repetition of the experiment at different PCTR loading arrangements with each material.

Except for the optimum neutron moderation values, the standard deviations quoted for a given quantity, M_o , in this report were calculated from

$$\sigma^2(\chi_o) = \sum_i \left[\frac{\delta M_o}{\delta M_i} \right]^2 \sigma^2(\chi_i) \quad (6)$$

where

$\sigma^2(\chi_o)$ is the variance in M_o ,

$\sigma^2(\chi_i)$ is the variance in M_i ,

M_i is the i^{th} quantity of which M_o is composed.

Optimum neutron moderation and the corresponding reactivity were determined from least-squares fitting of the experimental data. The standard deviations reported for these values are those of the fit only. However, they are larger in each case than the standard deviation observed for any individual point by the propagation of errors method.

CONCLUSIONS

The minimum ^{235}U enrichment required for criticality in homogeneous uranyl nitrate systems is approximately twice that required for UO_3 systems. Consequently, the existence of the nitrate radical in a uranium system is of considerable importance when establishing criticality safety guides. The experimental data presented establish this minimum critical enrichment for homogeneous uranyl nitrate at 2.104 wt % with a standard deviation of 0.010. This results in a lower limit of 2.07 wt % at the 99% confidence level.

Optimum neutron moderation for 2.14, 2.26, and 3.04 wt % enriched uranyl nitrate homogeneous systems occurs at H:U atomic ratios of 8.0 ± 1.0 , 9.3 ± 0.5 , and 10.5, respectively, with maximum corresponding values of k_∞ of 1.013, 1.04, and 1.145. For the 2.14 wt % enriched uranium, k_∞ values greater than unity occur for H:U atomic ratios between 6 and 10.5. For 2.26 wt %, this range increases to between 4 and 15, and for 3.04 wt % to between 2 and 31. Optimum neutron moderation for the minimum critical enrichment of 2.104 wt % occurs at an H:U atomic ratio of about 7.6.

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APPENDIX

Table A-I. Isotopic Composition of Uranium in Uranyl Nitrate.

Isotope	Isotopic Enrichment (wt %) ^a			
²³⁵ U	2.14 ± 0.005	2.26 ± 0.005	3.043 ± 0.008	
²³⁴ U	0.0109 ± 0.0002	0.0119 ± 0.0003	0.0169 ± 0.0004	
²³⁶ U	0.0171 ± 0.0002	0.0165 ± 0.0004	0.0119 ± 0.0003	
²³⁸ U	97.83 ± 0.005	97.71 ± 0.005	96.93 ± 0.01	

a. Determined by mass spectrographic analysis.

Table A-II. Experimental Data and Comments - PCTR Uranyl Nitrate Experiments

Experiment	Material		B: ²³⁵ U Atomic Ratio	$\rho_{\text{material}} - \rho_{\text{void}}$ (cents) ^a	Normalized Cadmium Ratios Gold Activation			Comments
	²³⁵ U Enrichment (wt %)	H:U Atomic Ratio			Test Cell Average	Boundary ^b	Boundary ^c	
6702-2	2.14 ± 0.005	3.49 ± 0.09	0.0 0.0052 ± 0.0005 0.0086 ± 0.0005	-5.439 ± 0.035 -5.357 -5.335 0	2.18	2.16	---	Buffer H:U = 3.49.
6702-3	2.14 ± 0.005	6.37 ± 0.14 6.01 ± 0.14 6.01 ± 0.14 6.37 ± 0.14 6.37 ± 0.14	0.0 0.0299 ± 0.0003 0.0299 ± 0.0003 0.0121 ± 0.0003 0.0025 ± 0.0012 ^e	+0.141 ± 0.035 -3.111 ^d -3.161 ^d -0.527 0	2.80	1.97	---	Buffer H:U = 6.36.
6702-4	2.14 ± 0.005	6.37 ± 0.14 6.37 ± 0.14 6.37 ± 0.14 6.01 ± 0.14 6.37 ± 0.26	0.0 0.0061 ± 0.0003 0.0121 ± 0.0003 0.0299 ± 0.0003 0.0024 ± 0.0018 ^e	+0.198 ± 0.035 -0.330 -0.500 -3.246 0	2.70	2.67	---	Comparison of these data with 6702-3 shows the results are relatively insensitive to neutron spectrum at the outer edge of buffer region.
6702-5	2.14 ± 0.005	10.34 ± 0.10 10.38 ± 0.10 9.81 ± 0.10 10.36 ± 0.10	0.0 0.0334 ± 0.0003 0.0556 ± 0.0003 -0.0060 ± 0.0110 ^e	-0.535 ± 0.04 -3.446 -5.246 0	3.35	3.03(*)	---	Buffer H:U = 10.36. One measurement is the result of a test cell with a low H:U in an over moderated system.
6702-6	2.14 ± 0.005	10.34 ± 0.10 10.38 ± 0.10 10.48 ± 0.10 10.40 ± 0.17	0.0 0.0334 ± 0.0003 0.0224 ± 0.0003 -0.0059 ± 0.0110 ^e	-0.942 ± 0.04 -4.962 -2.336 0	3.31	3.23(*)	---	PCTR loaded for a slightly harder spectrum than 6702-5.
6702-7	2.14 ± 0.005	10.34 ± 0.10 10.38 ± 0.10 10.48 ± 0.10 10.40 ± 0.17	0.0 0.0334 ± 0.0003 0.0224 ± 0.0003 -0.0055 ± 0.0107 ^e	-0.879 ± 0.113 -4.837 -2.266 0	3.32	---	---	PCTR loaded for a harder spectrum than 6702-6.
6702-8	2.14 ± 0.005	8.08 ± 0.1 8.08 ± 0.1 8.46 ± 0.15 8.47 ± 0.15 8.46 ± 0.15	0.0 0.0 0.0197 ± 0.0003 0.0302 ± 0.0003 0.0138 ± 0.0009 ^e	1.219 ± 0.015 1.205 -0.340 -1.597 0	3.04	2.96(*)	2.76	Low H:U test cell. Buffer H:U = 8.44.
6702-9	2.14 ± 0.005	8.08 ± 0.1 8.46 ± 0.15 8.47 ± 0.15 8.46 ± 0.15	0.0 0.0197 ± 0.0003 0.0302 ± 0.0003 0.0133 ± 0.0025 ^e	1.203 ± 0.015 -0.301 -1.908 0	3.17	3.09(*)	3.73	PCTR loaded for softer spectrum than 6702-8.
6702-10	2.14 ± 0.005	7.23 ± 0.10 7.23 ± 0.10 7.26 ± 0.10 7.07 ± 0.10 7.07 ± 0.10 7.11 ± 0.10 7.17 ± 0.17 8.08 ± 0.10 9.25 ± 0.10 9.25 ± 0.10 9.25 ± 0.10	0.0 0.0 0.0 0.0206 0.0206 0.0311 0.0060 ^e 0.0 0.0 0.0 0.0	0.547 ± 0.199 0.379 0.831 -1.482 -1.509 -1.996 0 1.233 1.331 1.337 1.240	2.94	2.90(*)	2.83	Buffer H:U = 7.24. Buffer H:U = 7.24. Same test cell as used in 6702-9. High H:U test cells in under-moderated system should yield too high a $\Delta\rho$.
6702-11 ^f	2.26 ± 0.005	8.25 ± 0.11 8.25 ± 0.11 8.25 ± 0.11 8.24 ± 0.11 8.25 ± 0.19 9.39 ± 0.11 9.39 ± 0.11 9.82 ± 0.11 7.26 ± 0.11 11.00 ± 0.11 11.77 ± 0.11	0.0 0.0 0.0208 ± 0.0003 0.0309 ± 0.0003 0.0479 ± 0.0004 ^e 0.0 0.0 0.0 0.0 0.0 0.0	5.027 5.026 2.880 1.761 0 5.334 5.327 5.381 4.375 5.305 5.973	3.04	2.88(*)	2.72	Buffer H:U = 8.39.
6702-12	2.26 ± 0.005	11.00 ± 0.11 11.29 ± 0.11 11.30 ± 0.11 11.20 ± 0.19 9.82 ± 0.11 11.77 ± 0.11 8.25 ± 0.11	0.0 0.0203 ± 0.0003 0.0305 ± 0.0003 0.0418 ± 0.0014 ^e 0.0 0.0 0.0	4.914 2.412 1.391 0 5.308 3.795 5.997	3.36	3.44(*)	3.17	Buffer H:U = 11.27. Measurement was not used because the flux was mismatched - see 6702-11.

a. The values of the effective delayed neutron fraction β_{eff} and of the neutron lifetime λ determined from PCTR measurements are 0.0064 and 10^{-3} sec, respectively.

b. Ratio measured at center of face on outside boundary of buffer region; those noted by (*) are 3.5 in. in from outer boundary.

c. Ratio measured on face of graphite directly opposite center outside face of buffer region; separated from face of buffer region by a void 2.5 in. thick.

d. These are duplicate measurements and indicate the precision of the results. It is noted that this sample had a lower H:U ratio than did the buffer zone.

e. This value was obtained by interpolation or extrapolation of experimental data.

f. During this experiment, a reactivity change of $+0.541$ cents was observed when the temperature of the reactor was 21.2°C higher than that of the test cell. When the temperature difference was $< 1^\circ\text{C}$, no correction to the observed reactivity was made.

Fig. A-I. Change in Water Content of Uranyl Nitrate Exposed to Room Atmosphere.

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