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A SYSTEM FOR MEASURING THE URANIUM
CONCENTRATION OF LIQUID EFFLUENTS
FROM THE ENRICHED URANIUM
RECOVERY FACILITY AT
THE OAK RIDGE
Y-12 PLANT

R. A. Gallman

January 1976

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OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

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Oak Ridge Y-12 Plant

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ABSTRACT

A real-time, on-line monitor has been developed which can measure the uranium concentration of enriched uranium liquid effluent. The monitor uses the thorium L X-ray activity from the decay of ^{234}U as the basis for the measurement. A sodium iodide/thallium-activated scintillation crystal, optically coupled to a photomultiplier, is used as the detector. Evaluation results reveal that the limit of error over the uranium concentration calibration range is less than 1 wt ppm, and the lower limit of sensitivity is 0.50 wt ppm. This monitor cannot be used on a recovery facility which contains fission products. The monitor is installed in the Oak Ridge Y-12 Plant enriched uranium recovery facility and has been operating satisfactorily for some time.

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SUMMARY

Enriched uranium has become valuable commodity—worth more than any of the precious metals. The Oak Ridge Y-12 Plant^(a) operates an enriched uranium recovery facility that is designed to reduce, to a minimum, any losses of this valuable material. This facility converts various forms of scrap into high-purity uranium metal.

Liquid effluents, which are discarded from this process, are retained in large holding tanks until a chemical analysis verifies that each tank has an acceptably low uranium concentration. However, an on-line, real-time monitor is needed that eliminates the need for the holding tank and the chemical analysis. The enriched uranium liquid effluent monitor (EULEM) uses the thorium L X-ray activity from the decay of ^{234}U to measure the uranium concentration of the discard streams of the enriched uranium recovery facility. Evaluation of its performance reveals that the uranium concentration limit of error is less than 1 wt ppm and that the lower limit of sensitivity is 0.50 wt ppm, but the monitor cannot be used on a recovery facility that contains a significant amount of fission products. The EULEM is now installed in the Y-12 Plant enriched uranium recovery facility and has been operating satisfactorily for some time.

(a) Operated by Union Carbide Corporation's Nuclear Division for the US Energy Research and Development Administration.

INTRODUCTION

Applications for quantities of ^{235}U enriched uranium have increased markedly in the past few years. As its use has grown, so has its value. Today, highly enriched uranium (93.0 wt % ^{235}U , 1.0 wt % ^{234}U) has a monetary value greater than any of the precious metals. As a valuable commodity, it has to have strict accountability. Also, as its use has grown, so have the health and environmental dangers. Its release, therefore, has to be closely monitored and controlled.

Fabrication, using highly enriched uranium, has been a function of the Oak Ridge Y-12 Plant for many years. Foundry operations, rolling mills, and machine shops constitute the major fabrication functions that are concerned with this material. To safeguard this material and to prevent the accumulation of large inventories of scrap, the Y-12 Plant operates an enriched uranium recovery facility which is designed to receive most forms of fabrication scrap and process it to a high-purity product. The enriched uranium recovery process consists of a preliminary chemical treatment, an organic purification process, production of uranium tetrafluoride (UF_4), and calcium reduction of UF_4 to uranium metal. The principal solvent for the process is nitric acid. A more detailed description of the enriched uranium recovery process and facility is reported elsewhere.^(b)

When the uranium concentration becomes too low to make recycling through the recovery facility economical, this effluent is discarded to a treatment plant for release to the environment. To ensure that excessive amounts of uranium are not being discarded, the effluent is placed in a holding tank and a chemical analysis is made. When the uranium concentration is determined, the effluent is either released or recycled. But, this procedure is time consuming, and the number of holding tanks is limited. An on-line, real-time monitor is needed which has adequate sensitivity to serve both as an accountability monitor and as an environmental monitor. The ideal on-line, real-time monitor should require little maintenance, be insensitive to background and operating conditions, and have a uranium sensitivity of at least 1 wt ppm. The purpose of this report is to describe the monitor that has been designed to meet these criteria and to discuss its performance.

(b) Barkman, J. R.; *A Chemical Recovery System for Safeguarding Unirradiated Uranium*, Y-MA-3582; Union Carbide Corporation-Nuclear Division, Oak Ridge Y-12 Plant, Oak Ridge, Tennessee; July 1, 1970.

MEASURING ENRICHED URANIUM LIQUID EFFLUENT

BASIC CONSIDERATIONS

The purpose of the enriched uranium liquid effluent monitor (EULEM) is to determine the concentration of highly enriched uranium in the effluent streams of the enriched uranium recovery facility at the Oak Ridge Y-12 Plant. This highly enriched uranium is nominally 93.0 wt % ^{235}U , 1.0 wt % ^{234}U , 0.5 wt % ^{236}U , and 5.5 wt % ^{238}U ; the natural abundance, half life, specific activity, and major photon energies of each isotope are listed in Table 1. Detection of the alpha activity from these isotopes is not feasible because of the extremely short range of alpha particles emitted in water, which is the liquid medium. The specific activity is calculated using the formula:

$$S = (0.693) \frac{A_0}{t_{1/2} M} \quad (1)$$

where:

S represents the specific activity in disintegrations per second per gram,

A_0 Avogadro's number (6.025×10^{23} atoms per mole),

$t_{1/2}$ the isotope half life in seconds, and

M the isotope mass number in grams per mole.

Table 1
PERTINENT FACTS ON THE MAJOR
URANIUM ISOTOPES

Uranium Isotope	Percent Natural Abundance	Half-life (yrs)	Specific Activity ⁽¹⁾	Major Photon Energies (keV)
234	0.0057	2.47×10^5	228.915	Th L X Rays: 53; 117
235	0.7196	7.10×10^8	0.079	Th L X Rays: 143; 185; 204
236	-	2.39×10^7	2.345	Th L X Rays -
238	99.276	4.51×10^9	0.012	Th L X Rays -

(1) Expressed as d/sec/ μg .

As is evident from Table 1, ^{234}U has a much higher specific activity than does ^{235}U ; in fact, it is nearly 3000 times greater. This comparison means that, in the highly enriched uranium, the ^{234}U activity is about 30 times greater than is ^{235}U . Since the count time must be kept to a minimum to approximate a real-time measurement, and since the ^{234}U concentration is directly related to the total uranium concentration, the photon activity from this isotope is the basis for the design of the EULEM. Figure 1 illustrates the decay sequence of ^{234}U . As indicated, it decays by alpha emission to the ground state of ^{230}Th 72% of the time and to an excited state of ^{230}Th 28% of the time. This excited state decays

to the ground state by either gamma emission or internal conversion, a process in which an electron is ejected from the atom. The L-shell internal conversion coefficient for thorium is 130, which indicates that decay is almost entirely by internal conversion. When the L-shell vacancy is filled, thorium L X rays are emitted. Since the major photon activity from ^{234}U is the thorium L X rays, the measurement technique is set up to detect these emissions.

EQUIPMENT

A schematic diagram of the EULEM is given in Figure 2. The enriched uranium liquid effluent activity is detected by a scintillation detector. The detector consists of a sodium iodide/thallium-activated [NaI(Tl)] crystal, 127.0 mm in diameter and 1.6 mm thick, which is optically coupled to a 127.0-mm-diameter photomultiplier. The detector has an energy resolution better than 36%, full width at half maximum (FWHM), at an energy of 22 keV.

Figure 1. DECAY SEQUENCE FOR URANIUM-234.

Figure 2. PRINCIPAL COMPONENTS OF THE ENRICHED URANIUM LIQUID EFFLUENT MONITOR.

The detector has a temperature operating range from -40 to 50°C . The NaI(Tl) crystal converts photons to light pulses, and the photomultiplier converts these light pulses into electrical pulses. The electrical pulses are then passed through a preamplifier and amplifier for pulse shaping and amplification. A pulse-height analyzer is used to establish an energy window around the thorium L X-ray peaks and provides an output to a digital ratemeter that is proportional to the activity within this window. The amplifier, pulse-height analyzer, and digital ratemeter are NIM-mounted modules and are powered by a nuclear instrumentation module (NIM) power supply. When the system is placed in service, the digital ratemeter output goes to an alarm circuit.

To improve the sensitivity of the measurement, natural background activity must be minimized. This type of activity is due to such sources as cosmic radiation, thorium and uranium in the soil and in buildings, and radon and thoron gases in the atmosphere. To minimize their effect, the detector and the material being evaluated must be shielded. Figure 3 indicates the elaborate shielding that is used. Note that it totally encloses the detector and material. Thus, a photon which does not originate inside the shielding to reach the detector must traverse 13 mm of stainless steel, 51 mm of lead, 1.6 mm of cadmium, and 0.8 mm of copper. The cadmium layer is necessary to attenuate the lead X rays; the copper is necessary to attenuate the cadmium X rays.

Figure 3. PHOTON SHIELDING THAT ENCLOSES THE SAMPLE RESERVOIR AND DETECTOR.

It is not possible for the EULEM to be connected directly to the waste stream line because this arrangement would require that the recovery facility be shut down for initial installation and thereafter for periodic maintenance. It would also complicate the shielding problem. Instead, a branch from the main line is constructed (Figure 2) which connects the effluent stream to the sample reservoir. The sample reservoir (Figure 3) contains a fixed volume of material [$1.219 \times 10^{-3} \text{ m}^3$ (1219 cm^3)] for the detector to evaluate. It is designed so that the detector measures a thickness of solution which is infinitely thick to the thorium L X rays, eliminating the problem of uranium buildup on the stainless steel surfaces. Beryllium is used as the window through which the thorium L X rays can pass to reach the detector because beryllium has a low attenuation coefficient at the thorium L X-ray energies. This property enables it to be thick enough to minimize the danger of rupturing without significantly decreasing the count rate. However, because beryllium is susceptible to corrosion by the waste stream, a Mylar sheet is used to cover its inner surface. To create a tight, noncorrodible seal between the beryllium window and stainless steel flange, Teflon gaskets are used. These gaskets must perform well since a leakage of enriched uranium solution could create health and criticality problems.

The enriched uranium recovery facility cannot be used to evaluate the EULEM because it would have to be shut down numerous times in order to introduce solutions of known uranium concentrations. To carry out the evaluation, a test loop was constructed (seen in Figure 4 along with the shielding) which can simulate the operating conditions of the

Figure 4. ENRICHED URANIUM LIQUID EFFLUENT MONITOR TEST LOOP.

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recovery facility. The main components of the test loop are: an electrical pump, an immersion heater, a flow ratemeter, valving to adjust the flow rate and pressure, and a reservoir to hold the solution. By using this system, connected to the sample reservoir, solutions of known uranium concentrations can be introduced to the EULEM. Also, the effect on the measurement of such operating parameters as temperature, pressure, and flow rate can be determined.

PERFORMANCE

Before any evaluation of the usefulness of the EULEM as an enriched uranium concentration gage is possible, several steps must be taken to ensure the quality of the results. First, the detector resolution must be measured, using standard radioactive sources, to make sure it is adequate. Second, the amplifier gain must be adjusted to separate the thorium L X-ray peaks from low-level noise. Third, a pulse-height analyzer energy window must be established around the thorium L X-ray peaks. The second and third steps are carried out by using a standard of enriched uranium that has been electrodeposited onto a metal substrate. Once the detector and its electronics have been optimized, the effectiveness of the shielding is studied. An energy-spectrum comparison of background without and with the shielding is provided in Figure 5. The energy window for the thorium L X rays is also shown. A shielded background spectrum and an enriched uranium spectrum are given in Figure 6.

Figure 5. COMPARISON OF THE BACKGROUND ENERGY SPECTRUMS WITH AND WITHOUT SHIELDING.

Figure 6. COMPARISON OF SHIELDED BACKGROUND AND ENRICHED URANIUM ENERGY SPECTRUMS.

The amount of background activity is important because it controls the lower limit of sensitivity. The background activity within the thorium L X-ray energy window is determined: (1) for a clean sample reservoir, both empty and water filled; (2) for a sample reservoir after an enriched uranium solution has circulated through it, both empty and water filled; and (3) for a sample reservoir which has been flushed with clean water after an enriched uranium solution has circulated through it, both empty and water filled. Data for this determination are reported in Table 2. The background activity rises sharply after the enriched uranium solution has been circulated through the sample reservoir, but it drops off after a single flushing with clean water. It is apparent that some enriched uranium remains on the interior walls of the sample reservoir after the flushing step. Fortunately, the Mylar sheet retains very little. Experience shows that the background activity with clean water remains at about 400 counts per 100 seconds, meaning that the lower level of sensitivity is that concentration of enriched uranium that will produce a count rate of about 600 counts per 100 seconds.

There are two operating parameters of the enriched uranium recovery facility that can affect the performance of the EULEM: (1) the temperature of the enriched uranium effluent ranges from ambient up to 95° C, and (2) the flow rate and line pressure of the main enriched uranium effluent stream can fluctuate. To evaluate these fluctuation effects, a solution of known uranium concentration was introduced into the test loop which could cause these two quantities to fluctuate. Results show no effect from either, even when fluctuations are created which are twice as large as those that occur in normal operation.

Table 2
BACKGROUND ACTIVITY IN THE
THORIUM L X-RAY
ENERGY WINDOW

Σ Window (cts/100/s)	
Empty	Water Filled
<u>Clean Reservoir</u>	
247	188
251	189
269	198
263	189
256	194
Average 267	192
<u>Enriched U Added to Solution</u>	
793	405
805	391
782	475
790	523
801	510
Average 794	461
<u>Enriched U Flushed from System</u>	
691	414
538	405
403	339
380	253
541	381
Average 512	358

Determining the temperature effect is more difficult. At temperatures significantly above ambient, a solution in the test loop evaporates faster, which can significantly alter the uranium concentration. The enriched uranium recovery facility is a closed system and has no evaporation problems. The temperature effect is determined by heating clean water in the test loop to the desired temperature and counting highly enriched uranium standards. Results on ten standards, counted at three different temperatures, are presented in Table 3. These data indicate that the EULEM is not temperature dependent up to an operating temperature of 93° C.

The sensitivity of the EULEM to highly enriched uranium is determined by introducing solutions of known uranium concentration into the test loop, circulating the solutions through the sample reservoir, and accumulating the counts for 100 seconds. The counting interval of 100 seconds is used to permit relatively short data-collection intervals while maintaining a suitable precision. Solutions of five different uranium concentrations and the observed count rate are reported in Table 4. The data from Table 4 serve as a calibration and are used to obtain a linear, least-squares-fit equation:

$$CR = 535.8(U) + 335.4 \quad , \quad \text{or} \quad (2)$$

$$U = (1.865 \times 10^{-3}) (CR) - 0.62 \quad , \quad (3)$$

where:

CR represents the observed counts per 100 seconds, and

U the uranium concentration in wt ppm.

The data of Table 4 along with Equation 3 are presented graphically in Figure 7. As a check on the reliability of Equation 3, solutions of known uranium concentration are counted, and the observed count rate is used in Equation 3 to find a

Table 3
OBSERVED COUNT RATE AT THREE
TEMPERATURES

Enriched Uranium Standard	Observed Count Rate at Three Temperatures (cts/100 s)		
	27° C	66° C	93° C
1	760	690	710
2	1,090	1,080	1,060
3	1,570	1,600	1,610
4	1,850	1,880	1,890
5	2,940	3,160	2,980
6	4,810	4,750	4,790
7	7,390	7,360	7,300
8	16,300	15,850	15,650
9	19,770	19,740	19,660
10	22,730	22,620	21,990

calculated uranium concentration. The calculated and known uranium concentrations are compared in Table 5. For the 15 different solutions, the maximum difference between the known and calculated uranium concentrations is 0.71 wt ppm. These results indicate that the least-squares-fit calibration equation has a limit of error which is less than 1 wt ppm over the calibration range. From Equation 3 it is determined that 0.50 wt ppm corresponds to 600 counts per 100 seconds, which is the lower limit of sensitivity of the EULEM.

Table 4
VARIATION OF OBSERVED COUNT RATE
WITH URANIUM CONCENTRATION

Uranium Concentration (1) (wt ppm)	Observed Count Rate $\pm 2\sigma$ (cts/100 s)
0.00	410 $\pm$ 40
1.35	960 $\pm$ 62
4.45	2,600 $\pm$ 102
10.20	6,000 $\pm$ 155
24.82	13,580 $\pm$ 233

(1) Determined by fluorometric analysis.

impaired. Maintenance of the EULEM can be divided into two categories: (1) that pertaining to the sample reservoir, and (2) that for the NaI(Tl) scintillation detector and its signal-processing electronics.

Sample Reservoir

The major maintenance problems associated with the sample reservoir are leakage, corrosion, and uranium buildup on the interior. Leakage of highly enriched uranium effluent can cause health physics and criticality problems as well as possibly corroding the detector. Eight bolts are used to retain the stainless steel/Teflon/beryllium/Mylar/Teflon/stainless steel seal. Over a four-month period and under a variety of conditions created by the test loop, no leakage has occurred; nevertheless, the seal is to be inspected whenever other maintenance is being carried out. Corrosion is a problem that can cause leakage and buildup. For this reason, the materials used to construct the sample reservoir are selected on the basis of their corrosion resistance (stainless steel, Teflon gaskets). The bery-

The enriched uranium liquid effluent monitor is now installed in the enriched uranium recovery facility and has performed satisfactorily since its installation.

MAINTENANCE

All equipment requires some type of periodic maintenance, and the EULEM is no exception. However, if routine maintenance requires the removal of any equipment from service for extended periods, its usefulness is significantly

Figure 7. URANIUM CONCENTRATION VERSUS THE COUNT RATE.

Ilium X-ray window is susceptible to corrosion, but the Mylar sheet protects it. Uranium buildup is important in that it affects the background activity. Buildup on the Mylar sheet is the only buildup that is significant since that which is on the stainless steel part of the sample reservoir does not contribute to the background count rate. During the four months that the EULEM has undergone evaluation, the background activity has increased from about 200 counts per 100 seconds for a clean, water-filled sample reservoir to about 400 counts per 100 seconds for a contaminated, water-filled sample reservoir. Although this doubling of the count rate is not considered excessive, part of the routine maintenance will be to disassemble and clean the sample reservoir periodically. This work requires that the EULEM be out of service for approximately four hours.

Scintillation Detector

The major maintenance problems associated with the NaI(Tl) detector and its signal-processing electronics are maintaining power to them and amplifier gain drift. The high-voltage power supply, which is stable against changes of line voltage, load, and temperature, and the NIM power supply are located in the same instrument cabinet. This arrangement makes it easy to periodically inspect the cabinet to make sure that both power supplies are functioning properly. Amplifier gain drift can be very serious because it can cause the thorium L X-ray peaks to occur outside the preset energy window. An enriched uranium standard is used to check for amplifier drift and to adjust the window, if needed. The standard is placed in front of the detector and counted. If the count rate is different from the standard count rate, the amplifier gain is adjusted to get the proper value; if amplifier gain adjustments do not give the standard count rate, then the energy window can be adjusted. This maintenance step, which is to be done every week, will keep the EULEM out of service less than one hour.

Table 5
COMPARISON OF CALCULATED AND MEASURED
URANIUM CONCENTRATIONS

Observed Count Rate (cts/100 s)	Uranium Concentration (wt ppm)		
	Calculated from Least-Squares Fit	Based on Chemical Analysis	Difference
470	0.26	0.36	- 0.10
660	0.61	0.68	- 0.07
700	0.68	0.72	- 0.04
1,140	1.51	1.39	+ 0.12
1,450	2.08	2.13	- 0.05
1,650	2.46	2.06	+ 0.40
1,810	2.00	2.91	- 0.11
2,020	3.15	2.73	+ 0.42
2,150	3.39	3.68	- 0.29
2,530	4.10	3.39	+ 0.71
4,860	8.42	8.20	+ 0.22
7,120	12.66	13.01	- 0.35
8,570	15.30	15.07	+ 0.29
9,670	17.41	17.82	- 0.41
11,050	19.99	19.94	+ 0.05

LIMITATIONS

There are four major limitations on the usefulness of the EULEM:

1. Applicability to a single enrichment level. The calibration curve in Figure 7 is only good for enriched uranium which is 93.0 wt % ^{235}U and 1.0 wt % ^{234}U . If the concentration of a different enriched uranium is to be determined, a new calibration is required. It also requires knowledge of the ^{234}U enrichment and the relationship between ^{234}U and

total uranium. In addition, if the enrichment level is such that ^{234}U drops below 1.0 wt %, the counting time must be increased to maintain the same limits of error.

2. Loss of sensitivity when fission products are present. Fission products produce a background continuum in the thorium L X-ray energy window, the intensity of which depends on the fission-product concentration. Low levels merely increase the lower limit of sensitivity, but concentrations which increase the background count to 2000 counts per 100 seconds or more seriously impair the ability of the EULEM to detect uranium concentrations of 5 wt ppm or less. Consequently, the EULEM cannot be used on an enriched uranium recovery facility which has an effluent stream that contains significant concentrations of fission products. Also, the EULEM cannot identify any other radioisotopes (such as fission products), since it is designed solely for the detection of uranium.
3. Viscosity changes of the effluent stream. If the viscosity is significantly variable, the attenuation coefficient of the solution is altered enough to affect the observed count rate, and that factor can change the calibration curve or the limits of error. The change in attenuation coefficient which corresponds to the normal change in viscosity has been calculated and has been found to have a negligible effect. In a facility that has greater viscosity changes, however, the performance of the EULEM is degraded.
4. Possibility of a 100-second delay before the alarm is sounded. This limitation is due to the 100-second count time needed to maintain a limit of error of 1 wt ppm of uranium. By comparison, a ten-second count time has a limit of error of nearly 5 wt ppm. The normal uranium concentration range of the effluent stream of the enriched uranium recovery facility is 5 wt ppm or less, so that a measurement has to have a limit of error significantly less than 5 wt ppm to be useful.

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