TOPICAL REPORT WIN-114

SURVEY AND PREVENTION TECHNIQUES FOR CONTROL
OF RADIOACTIVITY HAZARDS AT THE MONTICELLO
URANIUM MILL

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ABSTRACT

This report describes the techniques used in sampling, analyzing, and evaluating results for determining hazards from external radiation, airborne radioactive material, radioactive contaminants in mill effluents, and urinary uranium, as applied to the Monticello, Utah, uranium mill. Corrective measures used by National Lead Company, Inc., at the Monticello Mill are described along with the results from later surveys which showed large reductions in the potential radiation hazards. Details are given of the methods used to determine personnel exposures to radioactive and silicosis-producing dusts, potential external radiation hazards and in assaying mill effluents for uranium and radium, assaying urine samples for uranium, as well as the standard operating procedures issued at Monticello to employees for preventing and/or controlling personnel exposures.

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INTRODUCTION

In all commercial milling operations problems of waste disposal, air and water pollution, and safety of employees against potential hazards of the operation are present. State and federal laws have been legislated to insure the safety of both the general population and company employees. The processing of radioactive materials such as uranium requires particular attention to these safety measures. Early in 1957 the U. S. Atomic Energy Commission initiated Part 20, Title 10, of the Code of Federal Regulations (CFR) entitled, "Standards for Protection Against Radiation," to establish general standards for protection of licensees, their employees, and the public against radiation hazards.

The Health and Safety Laboratory of the U. S. Atomic Energy Commission made a radiation survey of the AEC owned mill at Monticello, Utah, in April of 1957 under the direction of W. B. Harris. At the time, National Lead Company, Inc., contract operator of the mill, was in the middle of an intensive campaign to improve operating conditions, eliminate potential safety hazards, and provide improved working conditions.

In June of 1958, the Raw Materials Division of the AEC requested National Lead Company, Inc. to conduct a radiation survey of the Monticello uranium ore processing mill.

During late June and early July of 1958, personnel from the Health and Safety Division, National Lead Company of Ohio, Cincinnati, Ohio, assisted by the staff of National Lead Company, Inc. at Monticello, conducted an industrial hygiene and radiation survey of the mill.^{1/} The study was aimed at determining and evaluating exposure sources rather than at individual personnel exposures in order to develop recommendations which would bring the personnel exposure within acceptable limits as defined by Part 20. In general, the recommendations were of two major types: (1) those that could bring about a significant reduction in personnel exposures through improvements in

1/ Industrial Hygiene & Radiation Survey of Monticello Mill, Monticello, Utah, J. P. Wing, Health & Safety Division, National Lead Company of Ohio, Cincinnati 31, Ohio, August 8, 1958

operating procedures, maintenance, housekeeping and minor equipment modifications, all requiring little or no capital investment; and (2) those that would require major modifications to existing equipment and addition of new equipment requiring some investment of funds but that would give a more fixed control over existing sources of personnel exposure. Personnel from the Raw Materials Development Laboratory groups in Winchester, Massachusetts and Grand Junction, Colorado, assisted by the staff of National Lead Company, Inc. at Monticello conducted a survey of pollution from the mill's liquid effluents. Many of the recommendations from this over-all survey have been put into practice at the Monticello Mill and additional proposals are under consideration.

SUMMARY

At the request of the Raw Materials Division of the U. S. Atomic Energy Commission, a radiation survey was made of the Monticello, Utah, uranium ore processing mill which is operated by National Lead Company, Inc., for the Commission. The survey, which was made during the months of July through October 1958, included measurement of external radiation, airborne radioactive dust, radioactive concentration in mill effluents, and a study of personnel internal exposures to uranium through a routine urine sampling program.

External radiation measurements indicated that radiation levels were well below the permissible dose limits as specified in the Code of Federal Regulations, Part 20, Title 10.

Several hundred dust samples were collected throughout the mill to evaluate airborne radioactivity levels. Samples were counted in both scintillation and proportional gas-flow counters for total alpha radiation. Based upon this maximum permissible concentration (MPC) as stated in Part 20, this survey indicated that the ore sample plant, crushing areas, sample preparation area, and yellow cake drying area contained airborne radioactive material sufficiently in excess of this level to be considered significant. Most of the other plant areas were below this permissible concentration. A plantwide program to reduce or eliminate sources of dust, to improve housekeeping, and to formulate and enforce standard operating procedures particularly designed for dust control, resulted in a material reduction of these dust levels. Efforts of the dust suppression program were largely centered on the ore crushing and yellow cake drying and barreling operations. Although in some areas airborne contamination may still exceed the MPC, the operators exposures are minimized by the conscientious use of respirators until such time as additional equipment for dust control can be installed.

A urine sampling and assaying program has been in effect at Monticello for nearly three years. Uranium in the urine is determined by a fluorometric assay procedure. Expansion and wider coverage of personnel by the program during the past few months has shown an encouraging steady decline of urinary uranium in samples from nearly all operators subjected to large quantities of dust. A reference level of 0.05 milligrams of uranium per liter (mgU/l) of urine is currently being used at Monticello for evaluating the significance of the data

obtained. Studies on urinary uranium have helped determine unsuspected areas of high airborne contaminations as well as pointing out operators who are possibly receiving significant amounts of uranium internally by inhalation and/or ingestion. All plant personnel are included in the urine sampling program. Some employees are scheduled only once a year; while others who are working in areas having higher air dust levels, are scheduled weekly.

Weekly samples of the tail pond water are composited from daily samples and are assayed for radium. Because radium is the one radioactive material whose permissible level as stated in Part 20 is grossly exceeded at every uranium mill where tailing solution has been assayed thus far, research work on treatment of mill effluents has been concentrated on reducing this isotope to specification levels. A survey of the acid - RIP circuit at Monticello showed only about 3 per cent of the radium in the ore was solubilized in the leach, and, following tails neutralization, only 0.05 per cent remained as soluble radium in the tailings released. However, this concentration was still 30 times the maximum allowable concentrations as listed in Part 20. The tailings from the Monticello alkaline RIP mill are varying from 270 to 2500 dpm of radium per liter, or approximately 30 to 280 times this level. Mill tests on treating mill pond effluents showed that 96 per cent of the radium could be adsorbed by pH adjustment and treatment by successive batch contacts with one gram of barium sulfate per liter of solution. Following additional laboratory research and mill tests, a process will be incorporated for treating the total effluent released from the Monticello Mill.

Other precautionary measures incorporated by Monticello to protect employees and the surrounding population included posting of areas of high airborne contamination and uranium storage areas, use of protective clothing in the concentrate handling area, requirements for certain operators to shower before leaving the plant, use of respirators in dusty areas, and regulatory measures regarding eating, drinking, and smoking.

As a result of the survey program and improvements made over the past few months, large advances have been made in the prevention and control of potential radiation hazards at the Monticello Mill.

EXTERNAL RADIATION

Permissible Dose

In Section 20.101, Paragraph (a), of Part 20 and Appendix A of that document, various maximum permissible doses are listed depending upon certain specified conditions. The lowest external radiation dose limit specified for the whole body is 300 mrem (millirems) per week. The rem, as used in Part 20, is a measure of the dose of any ionizing radiation to body tissue in terms of its estimated biological effect relative to a dose of one roentgen (r) of x-rays. One millirem is equal to 0.001 rem. In this survey, considering the source of the external radiation, one millirem was considered to be equivalent to one milliroentgen (mr) of X or gamma radiation for all practical purposes. For employees working 40 hours per week this would be equivalent to a dose of 7.5 mrem/hr. This figure was considered to be the maximum safe level with regard to the Monticello survey although under certain conditions higher dose levels are acceptable.

Survey Equipment

For purposes of the survey both portable alpha and portable beta-gamma survey instruments were available from the National Lead Company of Ohio plant. However, since alpha radiation from natural uranium does not generally present an external radiation hazard, the portable beta-gamma survey instrument is the most necessary tool in measuring external radiation for this type of evaluation. A battery operated Geiger-Muller (G-M) counter with a range of 0.1 to 20 mr/hr was used to measure the beta plus gamma and the gamma only radiation. The measure of one mr (milliroentgen) of gamma radiation as measured on this instrument was considered to be equivalent in dosage to one mrem.

Method of Radiation Survey

Mr. J. P. Wing, from the National Lead Company of Ohio, was in charge of the survey made in early July. A G-M counter was used to measure the radiation (gamma and beta plus gamma) at numerous points in the plant area and mill buildings. These included ore stockpiles, ore handling and processing area, sample preparation areas, and tailings ponds. Particular emphasis was placed on the yellow cake section of the plant. Most measurements were taken either 3 feet above a surface or 3 feet away from the material being measured. This was considered

to be a normal working distance for employees at these locations. In areas of process equipment, radiation measurements were taken 3 feet above the floor and 1 to 2 feet from the equipment. Spot checks of surface radiation levels were made in areas of higher radiation. In each area the high, low, and average radiation levels were measured and recorded.

Results of Radiation Survey

Based on a permissible dose of 300 mrem/week for an adult employee, the survey showed there was no hazard from external radiation (beta plus gamma or gamma only) to employees of the Monticello Mill in the normal performance of their work. Table I shows the levels of beta plus gamma radiation measured in various areas. The only significant levels in process areas were in the yellow cake section of the mill. The highest levels were 2.5 mrep/hr (beta plus gamma) found on one particular ore stockpile and in the yellow cake dryer enclosure. Average levels usually were in the range of 0.7 to 1 mrep/hr (beta plus gamma). The highest surface reading was 10 mrep/hr (beta plus gamma) and 3 mr/hr (gamma only) on one stockpile. There was no significant difference between the beta plus gamma and the gamma level only when measured 3 feet from the surface in the ore stockpile areas.

It should be pointed out that Part 20, Section 20.101, Paragraph (c), limits the permissible dose to a minor (under 18 years of age) to 10 per cent the exposure of other individuals. Employment of a minor in mill operations would require consideration as to his job assignment and his weekly exposure.

Where external radiation is a possible hazard, film badges are an effective method for determination of individual exposure to external radiation. Part 20 so states that anyone who receives or is likely to receive 25 per cent of the limits specified therein shall have appropriate personnel monitoring equipment supplied to him and be required to use it. Film badges cover this specification quite adequately.

Table I

External Radiation Exposure

(All readings in mr/hr, 3 ft above material)

<u>Location</u>	<u>High</u>	<u>Low</u>	<u>Average</u>
<u>Ore Stockpiles</u>			
South of plant	2.0	0.5	0.7 - 0.8
West of gate	1.0	0.5	0.6 - 0.7
Southwest of office	1.3	0.5	0.7 - 0.8
Shippers' ore lots	1.8	0.5	0.8 - 1.0
East of sample plant	2.5	0.5	0.8 - 1.5
<u>Tailings Ponds</u>			
New pond	0.8	0.5	0.6 - 0.7
Old ponds east of plant	0.8	0.5	0.6 - 0.7
Old ponds southeast of plant	1.3	0.5	0.7 - 0.8
<u>Process Areas</u> ^{1/}			
Yellow cake presses	2.0	0.6	0.8 - 1.0
Mud presses	1.0	0.6	0.7 - 0.8
Yellow cake dryer enclosure	2.5	0.6	0.8 - 1.0
Yellow cake barreling enclosure	1.0	0.5	0.6 - 0.8
Yellow cake drum surface reading	-	-	0.6
Yellow cake on floor grating, surface reading	-	-	1.0 - 4.0

^{1/} No significant level in process areas other than yellow cake area.

AIRBORNE RADIOACTIVE MATERIAL

Permissible Concentrations

Section 20.101, Paragraph (b) and Appendix B, Table I of Part 20 specify the maximum permissible concentration of airborne radioactive material to which individuals in a restricted area are to be exposed for 40 hours in any period of 7 consecutive days. Where the time of exposure is different than 40 hours, the limits should be adjusted proportionally. The limit established for natural uranium (soluble or insoluble) is 5×10^{-11} $\mu\text{C}/\text{ml}$ (microcuries per milliliter) of air which is equivalent to $110 \text{ dpm}/\text{m}^3$ (disintegrations per minute per cubic meter of air).

One common practice in monitoring airborne radioactive material in uranium milling operations is to collect airborne dust from a measured volume of air on a filter paper, determine electronically the alpha activity from the collected material, and relate these figures to the uranium limit noted in Part 20 on the assumption that all of the alpha activity found was from uranium. This procedure is certainly applicable to the milling operations involving the uranium concentrate (yellow cake). This procedure may also be used in the uranium ore handling areas to give a safe estimate of airborne radioactive material. Actually, only 25 per cent of the alpha activity of ore dust is due to natural uranium, and the other activity is due to the daughter products of uranium -238. However, if the total activity of uranium dust is less than $110 \text{ dpm}/\text{m}^3$, the specifications for natural uranium and the daughter products of uranium -238 will be met. For the purpose of the Monticello survey the conservative reference level of $110 \text{ dpm}/\text{m}^3$ was used.

A permissible limit has not been established for thorium -230, a relatively long half-life daughter product of the parent uranium -238 which is present in uranium ores where secular equilibrium exists or is being approached. Consideration may also have to be given to this isotope when counting dust samples, depending upon the level which may be defined as the permissible concentration.

Equipment for Collecting and Counting Dust Samples

The motor driven air sampling devices used in the survey had a capacity of up to 30 liters per minute and were equipped with a rotometer to measure air velocity and the appropriate fittings for connecting a sample holder for 1-1/8 in. diameter filter paper. Air sampling devices should be calibrated periodically to check the accuracy of the rotometers. Three pumps are generally used as will be described later. Filter paper should be 1-1/8 inch Whatman No. 41 or equivalent.^{1/} Prenumbered glassine envelopes were used for holding the filter paper after the sample was taken. Other miscellaneous equipment required included a stopwatch, extension cords (3-wire), tweezers, data sheets, and clipboard. The dust samples taken during the survey were analyzed at the National Lead Company of Ohio, using both gas-flow proportional and scintillation alpha counters.

During later follow-up surveys made by Monticello personnel after operational improvements had been made, counting equipment from the Winchester Laboratory was set up at Monticello. This equipment included a gas-flow proportional counter and a decade scaler. Accessory equipment included a lead shield, manual sample changer, preamplifier with a gain of 1 or 5, a cylinder of proportional counting gas, and a two-stage pressure regulator for maintaining an operating pressure of approximately 20 psig.

A calibrated alpha source standard is required for use with either type of counter to check periodically the geometry or operating efficiency of the instrument.

Although not required for measuring the amount of airborne radioactive material, additional equipment was used in the Monticello survey which included a modified casella cascade impactor for determining particle size distribution of dust, an air velocity meter for determining air velocities at ventilated openings, and pitot tubes with manometers to measure air velocities in ducts. In addition, samples were collected and analyzed to determine the extent of silica exposures.

^{1/} Smith, W. J., and Surprenant, N. F., "Media for Air Cleaning and Air-Assay Purposes," AECU-3119 (Oct. 3, 1955).

Dust Sampling Procedure

TYPE OF SAMPLES

Three general types of air samples were taken during the survey:

Process Sample: The process sample is collected to measure the amount of airborne radioactive contamination given off by a particular operation or piece of equipment. It is collected as close as possible (within a few inches) to the work or action portion of the operation. In the case of equipment, it is taken close to the visible or suspected source of dust.

Breathing Zone Sample: The breathing zone sample is collected within a few inches of the operator's nose for the purpose of determining the concentration of air dust which a person breathes in performing an operation. The breathing zone sample is also collected while a person performs work adjacent to a potential source or sources of airborne contamination.

General Air Sample: The general air sample is collected in a room or area to measure the amount of airborne radioactive dust to which the people who occupy the room are exposed. The source of contamination may or may not be associated with what the people are doing in the area. Where general air samples are collected, the airborne concentration should be expected to remain fairly constant while the samples are being collected. The air sampling devices should be located approximately five feet from the floor to simulate the approximate level of the operator's face.

PRELIMINARY OBSERVATIONS

Prior to sampling an area, operations should be observed for any factors which might cause abnormal dust samples. Such factors should be noted as dust collectors being off, broken dust collector bags, no water on in wet-type collectors, holes in vent tubes, and whether windows and/or doors are open or closed. Operations should be observed and notes made of what machinery is in operation in the area, maintenance work being done, and type of material being handled, such as dry ore or wet ore. When time studies are to be applied--in addition to breathing zone sample results--it is necessary to observe the operator's movements closely. Complete data are very important in this case. Note

what the operator is doing, how long it takes to perform various operations, time spent in other locations, time that operating equipment is off, etc.

Two approaches can be made in determining airborne dust exposures. Using the results of process, breathing zone, and general air samples the areas and particular phases of operations where conditions of potential overexposure exist can be pinpointed and, in some cases, an approximate exposure determined. Corrective measures can then be taken in an attempt to reduce the airborne dust in these locations to the maximum permissible level (110 dpm/m³ in the case of this survey.) This procedure would insure no overexposure even if the operator was in one particular area for 40 hours a week. The other approach is to study the operator's exposure using a breathing zone and general air sample results together with time studies. Emphasis can then be placed on improvements in particularly dusty operations. Follow-up dust samples may then show that even though some areas of overexposure may exist, the operator is not in that location for long enough periods of time to result in his being overexposed on a daily weighted exposure basis.

A daily weighted exposure is determined as follows:

1. List in a column all of the individual operations an employee performs in the course of a normal day's work, including such things as rest periods, lunch, etc.
2. Study these operations with respect to the time required for each. Record the time, as accurately as possible, in a column next to the list of operations described above and with corresponding times.
3. Collect breathing zone and/or general air samples for all of these operations, whichever is applicable. At least three samples of each type and of each operation is the minimum usually required.
4. Following sample analysis, record in three additional columns the high, low and average result of each operation sampled.

5. In another column, parallel to the earlier columns, record the value obtained by multiplying the average concentration found times the time spent for each operation and total this column.
6. Divide the value found as a total of this last column by the total time the employee works during the day. (This time should be the total of the times recorded for each operation.) The value obtained will correspond to the average exposure throughout the normal work day.
7. Dividing this last value by whatever reference value (MPC) in the same units that is applicable will result in a daily weighted exposure value. If this number is less than unity (1), this could be considered as a reasonable indication that the employee's exposure is not exceeding the reference level based on a daily weighted exposure or an exposure determined by sampling all operations performed and determining the over-all average exposure.

Prior to the Monticello survey, it was obvious that very dusty conditions existed in some areas while others were relatively clean. The dust survey made by J. P. Wing and J. E. Bailey consisted largely of general area samples with plans for breathing zone and operator time studies to be made at a later date after the worst dust sources had been eliminated.

SAMPLING

Most of the dust samples were taken at a rate of 20 l/min. This figure is approximately the amount of air an average size man working at a normal rate would take into his lungs each minute. Flowrate is adjusted and read on the rotometer. The rotometer reading should be checked frequently during the sampling period. The air quantities, or flowrates, were recorded in cubic meters per minute ($20 \text{ l/min} = 0.02 \text{ m}^3/\text{min}$). This simplifies the calculations to be made later, depending upon the reference level used, when the sample is analyzed. The time of sampling varies with dust conditions. For highest efficiency in counting samples, only a very thin layer of dust should be collected on the filter. When the filter paper is removed from the retaining ring, if a conspicuous ring can be seen, differentiating between the filter area in the middle and the clean area around the outside, there is probably too much dust on the filter. A duplicate sample should

be taken using a shorter sampling period. On many properly taken samples the dust is not easily visible. For process and breathing zone samples, times between 0.5 and 10 minutes are normally used; 2 to 5 minutes is usually the most desirable. For general air samples the time may vary up to 20 or 30 minutes depending on the air dust concentrations. A general air sample of 15 minutes duration is average.

Figure 1 is a sketch of part of the Monticello Mill showing the general areas sampled. Three air sampling devices were used simultaneously and were located in the vicinity of the operator's normal working positions. Approximate locations of the three sampling devices in each of the general areas sampled are shown in Figure 1 through 4 of the Appendix. Triplicate samples were taken with each of the three sampling devices resulting in nine samples in each particular area.

After the samples have been taken, the filter paper is removed from the sampling head with tweezers and placed in a prenumbered glassine envelope with the exposed side facing the number. The number of the sample is recorded on a sample data sheet together with a complete sample description. Two examples of data sheets are shown in Appendix Figures 5 and 6. Other pertinent information required on the data sheet is entered. In the event the sampling procedure is seriously disrupted, such as by stoppage of machinery, the samples for that particular phase of the operation are discarded and a new set of samples is collected when conditions are normal. Some special precautions used at Monticello in dust sampling and details related to care of equipment are listed in Section 1 of the Appendix.

COUNTING DUST SAMPLES

Details of counter and scaler operation can be obtained from the manufacturer's instruction books. The following counting procedures are general information considered to be good laboratory practice. Alpha counting of the dust samples can be done directly as long as the quantity of dust on the filter paper is kept low. The ore dust samples should be allowed to stand for 6 hours before counting to allow for decay of most of the daughters of radon gas which may have been present in the air and adsorbed on the filter paper. In cases where the count shows the radioactivity to be many times over or conservatively under the permissible concentration, the error due to radon daughters is relatively unimportant.

Figure 1
Location of Dust Sampling Areas
Monticello Mill

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A standard alpha source (in the range of 3000 dpm is convenient) is used daily to determine the efficiency or geometry of the instrument by counting for 10,000 counts. Three such checks at the beginning of the day insure dependability. The background is determined by counting a blank planchet and ring in the chamber. This should be done overnight at least once a week and any other time the counter is not in use. A high background may be due to contamination and the counting chamber should be cleaned according to the manufacturer's instructions. When counting high dust levels, the background should be determined often to check for contamination of the chamber.

The filter paper is removed from the envelope with tweezers and placed on the sample holder. The retaining ring is placed over the paper to hold it in place and the sample is then inserted into the counting chamber. Dust samples should be counted for 25 minutes or 100 counts, whichever comes first, or with a minimum time of 2 minutes. Some laboratories find the accuracy obtained by counting to 32 counts (binary scaler) or 15 minutes, again - whichever comes first - to be sufficiently accurate.

The following calculations are used in counting procedures for dust samples:

Background (cpm) = counts/time in minutes

Geometry (%) = $\frac{\text{cpm obtained from standard} \times 100}{\text{Known disintegration rate (dpm)}}$

Counts per minute for sample = $\frac{\text{total count}}{\text{total time, min.}}$ - background

Disintegrations per minute, dpm = $\frac{\text{cpm}}{0.7 \text{ adsorption factor}} \times \frac{100}{\% \text{ geometry}}$

The adsorption factor of 0.7 for filter paper is an approximation generally used for alpha counting of dust. ^{1/}

Dpm/m³ = $\frac{\text{dpm}}{\text{rate of sampling, m}^3/\text{min} \times \text{time of sampling, min}}$

^{1/} Alercio, J. S., and Harley, J. H., "Evaluation of Alpha-Particle Absorption by Filter Paper," Nucleonics, Vol. 10 (November 1952).

An evaluation is presently underway at the Winchester Laboratory to determine the feasibility and reliability of assaying by the fluorometric method uranium ore and concentrate dust collected on the filter paper. This would eliminate the necessity of counting equipment for dust samples, although the fluorometric method may be more costly and is definitely slower.

Results of Dust Survey

The results of the dust sampling program showed that airborne radioactivity in some areas of the sample plant, crushing plant and yellow cake drying and packaging areas was from 2.0 to 78 times the Part 20 maximum permissible concentration. Other process areas were well below the Part 20 maximum permissible level. Results of the dust survey are shown in Table II. As can be seen, a number of these levels were of sufficient magnitude to warrant immediate corrective action.

The air dust levels found in the majority of the areas of the RIP Building were believed to be due largely to radioactive material becoming airborne from the yellow cake drying and barreling section, and either from natural convection or from outside drafts, being carried to adjacent areas of the building.

Table II
Airborne Radioactive Material

<u>Location</u>	<u>No. of Samples</u>	<u>Alpha, dpm/m³</u>		<u>Avg.</u>	<u>X MPC of ^{1/} 110 dpm/m³</u>
		<u>High</u>	<u>Low</u>		
<u>Sample Plant</u>					
Sample preparation bldg.	5	1884	118	663	5.9
Bucking room	9	29	4	12	0.1
<u>Plant Crusher</u>					
Upper floor	9	3407	209	1226	11.5
Lower floor	9	16133	3073	8551	77.7
Conveyor area	9	550	4	105	0.9
<u>Acid Grind - Leach Building</u>					
Transfer point, feed conveyor	6	71	14	35	0.3
Ball mill feed point	6	34	12	21	0.2
Ball mill area	9	13	2	8	0.03
Leach tank area	9	87	11	29	0.3
Sand-slime separation area	9	30	3	12	0.1
<u>Acid RIP Building</u>					
Basket area	14	2950	10	327	2.9
Make-up tank area	1	-	-	41	0.4
Precip. tank area	6	365	42	230	2.1
Precip. tank, 2 ft. inside	9	968	22	276	2.5
Yellow cake press area	12	442	7	65	0.6
Yellow cake scrubber area	9	10218	112	3200	28.5
Yellow cake dryer area	9	335	39	225	2.0
Yellow cake barreling area	9	2927	73	1083	9.8
Cake weighing and storage area	9	67	8	33	0.3
<u>Sample Bucking Building</u>					
Yellow cake sample room	9	57	2	24	0.2

1/ Average alpha count divided by the reference maximum allowed concentration of 110 dpm/m³.

Table II (Cont'd)
Airborne Radioactive Material

<u>Location</u>	<u>No. of Samples</u>	<u>Alpha, dpm/m³</u>		<u>Avg.</u>	<u>X MPC of 110 dpm/m³</u>
		<u>High</u>	<u>Low</u>		
<u>Alkaline Plant</u>					
Ore bin area	9	67	8	33	0.3
Ball mill area,					
Upper deck	9	12	0	3	0.03
Lower deck	9	22	0	7	0.07
Tank room					
Near foreman's desk	9	41	1	14	0.1
Above thick. #1 & 2	9	39	2	8	0.1
Above thick. #3 & 4	9	18	2	8	0.1
Agitation tanks	9	26	0	6	0.05
Mill solution tank	9	41	1	14	0.1
Filter area	9	11	3	7	0.1
Precipitation tank area	9	183	4	59	0.5
Yellow cake press area	9	15	0	5	0.05
Yellow cake drying area	9	1098	193	554	4.9
Yellow cake barreling area	18	696	17	132	1.2
<u>Miscellaneous</u>					
Machine shop	18	16	2	8	0.1

The Industrial Hygiene and Radiation Department of the Health and Safety Division at the Feed Materials Plant, as a result of the dust survey, made numerous recommendations for improving conditions at the Monticello Mill.

Plant Improvement to Reduced Exposure

With relatively small capital expenditures it was possible to reduce greatly the amount of airborne radioactivity. Major equipment improvements such as installing new dust collectors for the plant crusher building and yellow cake drying and drumming area have been proposed.

In the ore sampling plant water sprays were installed to dampen the ore as it is dumped from trucks into the hopper. Hooding was placed around the primary crusher so that approximately 65 per cent enclosure was obtained. This hooding was connected to the dust collection system. Aprons and panels were installed around the feed chute and the bottom of the primary crusher to confine dust. The sample splitter following the primary crusher was completely covered and the access door gasketed to contain dust. A complete enclosure was built around the double decked vibrating screen and secondary crusher because hooding or enclosing each individual component was impractical. The enclosure was then connected to the dust collection system.

In the plant crushing area, water sprays were installed to wet the coarse ore on the feeder, the product passing the hot-wire screen, and the product being discharged from under the crusher. The entire lower part of the impact breaker was enclosed, but capacity of the dust collection system did not permit connection to this enclosure. An additional enclosure was built around the hot-wire screen to include the crusher feed hopper and the enclosure was connected to the dust collection system.

The yellow cake drying and barreling enclosure formerly consisted of three levels with open grate floors. The three levels included a wet scrubber on top, the drum dryer below, and the barreling level on the lower floor. The enclosure was not tightly sealed and had canvas drapes at the doors. Natural ventilation due to heat from the dryer resulted in dispersion of fine product dust throughout that area of the building as is evident from the dust counts in the RIP Building shown in Table II.

The enclosure was tightly sealed, solid steel doors with gaskets were installed, and the enclosure was connected to the small ventilation system with the associated scrubber. The open grating was replaced with solid decking which stopped natural drafts and also permitted efficient hosing of the floors in the various levels.

The bulk of the basic engineering and design work for these modifications was accomplished at Monticello by an engineering-draftsmen team headed by C. H. Stevens, Engineering Division, National Lead Company of Ohio.

A general housekeeping improvement program and management directives for continued good housekeeping aided greatly in reducing the sources and/or accumulation of dust throughout the entire plant. Operating equipment and structural steel was cleaned and painted. Points of frequent spills were eliminated by changes in process piping. Operating procedures concerned with dust suppression, general housekeeping, use of respirators, and control of smoking, eating, and washing were revised. These Standard Operating Procedures are listed in the Appendix, Sections 2 through 6. It should be noted that in these Standard Operating Procedures the use of compressed air and brushes or brooms for cleaning equipment and surfaces is mentioned. This is poor practice. Proposed vacuuming equipment, when available, should eliminate the present necessity of cleaning by compressed air or brooms in certain locations. Wet cleaning methods are also satisfactory in some applications.

The combined effects of equipment modifications, improved housekeeping, and careful operating procedures have greatly reduced the airborne radioactive material as is shown in Table III and in Figures 1 through 4 in the Appendix. For comparison purposes, the results of a survey, which was made by the Health and Safety Laboratory of the U. S. Atomic Energy Commission in April of 1957, are shown in Figures 1 to 4, along with the results of the two surveys made by the National Lead Company. During the survey made by the Health and Safety Laboratory, wet ore was being treated and dust conditions in the crushing plant were relatively low.

The figures in the second column of Table III show the level of airborne radioactive dust from a survey made in October by J. E. Bailey after the many changes in the plant had been made. The figures in the first column are from the dust survey made in July. Very large reductions in the amount of airborne radioactive material are shown. The pronounced drop of the dust levels in the areas in the RIP building, other than within the product drying and barreling enclosure, show the effectiveness of containing the dust within the enclosure. The October survey verified original thinking: that the source of airborne radioactive material must be from the product drying and barreling section, not the RIP, precipitation, and filter areas where pulp, solution, and wet cake made up the process streams. It is

Table III
Comparison of Airborne Radioactive Material
Before and After Equipment Changes

<u>Location</u>	<u>X MPC of 110 dpm/m³</u>	
	<u>Before^{1/}</u>	<u>After^{2/}</u>
<u>Plant Crusher</u>		
Upper floor	11.5	1.3
Lower floor	77.7	6.8
Conveyor area	0.9	1.4
<u>Sample Plant</u>		
Sample preparation bldg	5.9	0.2
Bucking room	0.1	0.4
<u>Acid RIP Building</u>		
Basket area	2.9	0.2
Precip. tank area	2.1	0.5
Yellow cake press area	0.6	0.4
Yellow cake scrubber area	28.5	5.3
Yellow cake dryer area	2.0	14.3
Yellow cake barreling area	9.8	8.4
Cake weighing and storage area	0.3	0.9

1/ Survey by National Lead Company of Ohio, July 1958

2/ Survey by National Lead Company, Inc., October 1958

obvious from the October survey that additional dust collection equipment is required for the drying and barreling enclosure as well as the ore crushing areas.

Continuation of Dust Sampling Program

Continued dust survey work is now being performed by Monticello personnel. General air samples are to be collected semi-annually during the months of July and January to obtain representative samples during different seasons. Ore being processed during the summer is relatively dry while the feed is often wet during winter months.

General air samples are also to be collected anytime mechanical changes are made in processes or operations that may increase or decrease the amount of airborne dust in any area or location. A minimum of three separate sets of samples taken with three air pumps are to be collected in the area under observation. These three sets of samples will be taken over a period of three consecutive weeks.

Whenever excessive airborne dust exposures are indicated by high levels of uranium in urine samples, or when excessive dust is observed because of leaks in equipment such as the impact breaker, yellow cake dryer, etc., a minimum of three process air samples will be collected to locate the source and magnitude of the dust concentration. Three separate sets of general air samples will be taken in conjunction with the process samples to determine the exposure to operating personnel.

Breathing zone samples are to be taken in areas expected to be of medium or high exposure during each six-month period. These samples are to be taken in conjunction with general air and process samples to determine the source of the radioactive dust being inhaled or ingested when the quantity of uranium in urine samples is found to be above acceptable limits.

Plans are underway to investigate airborne radioactive levels on off-site or unrestricted areas adjacent to the plant. Stack sampling procedures are presently being studied. A perimeter air sampling survey is being considered depending on availability of suitable portable equipment. The use of permanently located fallout trays may also prove to be useful in determining the extent of off-site radioactive fallout.

URINE SAMPLING PROGRAM

Use of Urinary Uranium Assays

The use of uranium assays on samples of urine from plant employees has proven to be an effective indication of which employees are receiving highest bodily intake of uranium. Many different phases of the entire uranium industry, from the mine to the fabrication of fuel elements, are using urinary uranium assays as an aid in determining overexposure to individuals, locating unsuspected sources of radioactive dust, isolating operations which may be more conducive to ingestion of uranium, evaluating exposure with regard to various classifications of employees, and in studying the personal on-the-job habits of individual employees which affect his inhalation or ingestion of uranium.

While different authorities vary somewhat in their recommended maximum levels of uranium in urine, most of the control limits used are in the range of 0.025 to 0.05 mg of uranium per liter of urine. For the Monticello survey the maximum reference point used was 0.05 mg U/l. Because of the different biological effects in various individuals, in their ability to reject uranium and the differences in rate of rejection, urine sampling programs are generally designed to obtain samples at the end of the day before an employee leaves work for two or more days off and again at the time the employee returns to work. The off-shift sample will tend to show the highest level of uranium accumulated in the body, while the on-shift sample after two or four days off the job will show the rate of rejection by the individual. At high exposure levels, there will tend to be a large difference between off- and on-shift samples which will not be evident in employees working in areas of lower exposure.

When significant uranium contamination in urine becomes evident, investigations should be made as to the cause. It will generally be found that a lack of adherence to standard operating procedures; i.e., use of respirators, caution with regard to eating, smoking, and drinking, washing or showering before eating or leaving the plant, etc., will produce small amounts of uranium in the body, and hence in the urine.

Equipment Required

Special shower facilities, preferably in the near vicinity of the safety office or first aid room, are very helpful for obtaining

uncontaminated urine samples on a routine basis. No special analytical equipment is normally required at uranium mills as the assays can be made using the standard fluorometric procedure used in nearly all uranium mills. Miscellaneous equipment required includes a supply of new 4-ounce glass bottles with screw caps, identification labels, hydrochloric acid, medicine droppers, and data sheets or charts for recording results.

Procedures for Urine Sampling and Assaying

The urine sampling program at Monticello can be divided into three general categories; (1) Periodic samples from all employees, (2) The collection of regular samples from employees working in areas where the possibility of intake of uranium by ingestion and/or inhalation is highest, and (3) Spot check sampling of employees where the body intake could be significant depending on personal habits and operating conditions.

All Employee Samples

At least once a year, two urine samples are obtained from every employee, one near the end of the shift before leaving the plant for two or more days off and one when the employee returns to work after his days off. This sampling program is usually completed during a one- or two-week period. The purpose of these samples is to obtain a general record of every employee for future reference and comparisons in addition to being a general safeguard for all employees in the event uranium contamination may be present in unexpected locations.

Regular Samples

Continuous urine samples are obtained from all employees working in areas where airborne dust is likely to be higher in concentration or where the possibilities of ingestion of uranium are greater. Employees covered in this category are yellow cake dryer operators, product barreling operators, press operators, precipitation operators, crusher operators, sample buckers, and all helpers and utility men regularly employed in such locations. These samples are taken every week before leaving for days off and after returning. The purpose of these samples is to provide a margin of safety for those employed in locations where ingestion and/or inhalation of radioactive material may constitute a hazard to the employee in

case of defective equipment, poor personal hygiene, or any other condition that may deviate from standard operating procedures for that particular area.

Spot Check Samples

Spot check urine sampling is done on a continuous basis with a new group of operators following the completion of a previous group. Specific groups in this category are: RIP operators, maintenance men, truck drivers, warehouse men, tail pond operators, and general utility men. A short series of four samples (two sets of on- and off-shift samples) over a 2-week period is collected from each group. In the event two or more urine analyses from an individual show a uranium content of 0.05 mg/l or higher, a long series of 20 samples over a 10-week period is taken. In addition, an investigation is made of the working conditions and personal hygiene habits of the employee. The purpose of the spot check samples is to find any individual who may be inadvertently receiving undesirable amounts of radioactive materials.

Sampling Procedures

The cooperation of the employee and his supervisor is important in urine sampling to prevent the employee from forgetting to report for the sample. One break in the frequency of the sampling disrupts the evaluation of that series of samples. After reporting to the safety office, the employee showers and dries. The sample is then collected and 2 to 4 ounces of the sample transferred to a 4-ounce bottle. From 5 to 10 drops of HCl are added to the sample to prevent any precipitation; the bottle is labeled and sent to the laboratory. At the same time, pertinent information is recorded on a data sheet for future use.

The fluorometric analytical procedure is the same as has been reported in the literature 1/ 2/ except that a 0.2 ml aliquot of the urine sample is pipetted directly onto the sodium fluoride -

1/ Centanni, F. A., Ross, A. M., DeSesa, M. A., "The Fluorometric Determination of Uranium," WIN-34, February 10, 1956 and Anal. Chem. 28, 1651 (1956).

2/ Centanni, F. A., and Morrison, T. J., Jr., "Improvements in the Fluorometric Determination of Uranium," WIN-63 (April 3, 1957).

lithium fluoride pellet. A brief summary of the procedure is given in Section 7 of the Appendix.

Continuous records should be kept for all employees. For those employees whose urine analysis is followed weekly (regular samples), a graphic record plotting the assays of their off-shift sample and on-shift sample against the date is very useful and provides a source for quick reference.

Evaluation of Results

A more complete program of urine analyses was initiated at Monticello at the time a major effort was made toward reducing radiation exposure. The urinary uranium history of a number of employees since 1956, however, was helpful in establishing the expanded program. Only a few of the more demonstrative results from the program are included in this report. Table IV shows the history of five yellow cake dryer operators over a period of 5 months during which time mechanical improvements were made in the area as well as more rigid enforcement of standard operating procedures regarding smoking, eating, washing, etc.

It is very obvious from Table IV that intake by inhalation and/or ingestion was materially reduced over the 5-month period. It is also interesting to note the difference in the uranium content in the urine from five employees performing the same operations. The large difference in Employee "E" compared to the others could be due to personal habits or to different biological effects.

The urinalysis chart, Figure 2, shows the result of spot check samples on various groups of employees. The chart represents approximately 400 urine samples. Several of the points on the chart are for only a single sample. However, in classifications showing levels above the reference line each figure is a composite average of all the operators in the particular classification for the month. In these cases, each value is the average of from 4 to 20 samples. The results of repeat samples taken several months apart are shown in some classifications. Off-shift and on-shift samples show the effect of body rejection during the employees' time off. Operators in some areas where airborne radioactivity was known to be high, such as crusher operators, grizzly men, and sample buckers, were below the reference point of 0.05 mg/l. This was no doubt due to protection from conscientious use of respirators. The high level for the RIP operators and helpers, who do not wear respirators, was probably due to concentrate dust

Table IV

Urinary Uranium Analyses for Yellow Cake Dryer Operators

<u>Employee</u>	<u>Sample</u>	<u>Monthly Average, mg U/l urine</u>			
		<u>May</u>	<u>July</u>	<u>August</u>	<u>Sept.</u>
A	Off-Shift	0.320	0.213	0.111	0.072
	On-Shift	0.097	0.053	0.032	0.017
B	Off-Shift	0.477	0.196	0.135	0.088
	On-Shift	0.042	0.060	0.036	0.051
C	Off-Shift	0.231	Reclassified 0.066	New Assignment 0.019	-
	On-Shift	0.050	0.046	0.023	-
D	Off-Shift	0.459	0.268	0.121	Reclassified 0.068
	On-Shift	0.036	0.039	0.017	0.042
E	Off-Shift	1.000	0.407	0.187	0.152
	On-Shift	0.110	0.032	0.023	0.021
F	Off-Shift	-	-	-	0.025
	On-Shift	-	-	-	0.021
Average	Off-Shift	0.497	0.230	0.139	0.081
	On-Shift	0.067	0.046	0.027	0.030

in the area coming from the adjacent yellow cake drying section. Enclosure of the dryer section, which has been completed only recently, should have lowered the exposure to these operators as there was an appreciable decrease in the dust count in the area. The level of assays for the yellow cake dryer operators shows a downward trend. Uranium intake by these operators could be a combination of inhalation and ingestion.

The two graphs in Figure 3 show the variation in the assay of urine from two operators in the product handling section of the plant. Note the similarity between the on-shift and off-shift graphs. Reassignment in the case of Operator B reduced the high level of urinary uranium. In the case of Operator A, more strict operating procedures and compliance thereof was probably the reason for the downward trend and also for the sudden drop in the graph for about the 30th week of the year. These charts show the usefulness of continued sampling and plotting of assays for individuals working in areas of known contamination.

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Figure 3
Urinary Uranium Chart for Two Product Operators
Monticello Mill

MILL EFFLUENT SURVEY AND TREATMENT STUDIESPermissible Concentrations

Section 20.103, Paragraphs (a), (b), and (c) and Appendix B, Table II, Column 2, of Part 20 specify maximum permissible concentrations of certain radioactive isotopes which may be released into water in any unrestricted area. "Determinations as to the concentration of radioactive material shall be made with respect to the point where such material leaves the restricted area." Section 20.302 describes the "Method for obtaining approval of proposed disposal procedures" other than as authorized in Part 20 (quotations from Part 20). A portion of Appendix B, Table II of Part 20 which applies to the concentration in effluents released, is reproduced in Table V.

Table VPermissible Concentrations in Water AboveNatural Background

<u>Material</u>	<u>$\mu\text{c/ml}^{1/}$</u>	<u>dpm/l</u>
Ra 226 $\pm$ 1/2 dr	4×10^{-9}	8.8
U-natural (soluble)	7×10^{-6}	15,400 or 0.01 g U/l
Th-natural (soluble)	5×10^{-8}	110
Unidentified alpha emitters or any undetermined mixtures of alpha emitters	1×10^{-7}	220

1/ Figures from Part 20. $\mu\text{c/ml} \times 2.22 \times 10^9 = \text{dpm/l}$

The maximum permissible concentration specified for natural uranium, which would include U-238, U-235, and U-234, is 7×10^{-6} $\mu\text{c}/\text{ml}$ of water, equivalent to approximately 0.01 g U/l of water. The Ra-226 specification of 4×10^{-9} $\mu\text{c}/\text{ml}$ or 8.8 dpm/l is one of the most stringent; and for this reason, radium is one of the isotopes of most concern in effluent disposal.

Analytical Techniques

Fortunately the maximum permissible concentration for uranium is within the range of the fluorometric method commonly in use in most all uranium mills.

Although several analytical methods for radium are in use, the Winchester group felt these were not suitable for use in uranium mills because they (a) required elaborate and sensitive equipment, (b) were too long or, (c) were not accurate enough. Therefore, a carrier-free separation of radium was developed. Details of this procedure are given in Section 8 of the Appendix. The method uses a lead sulfate carrier precipitation and an ion exchange separation of lead from radium followed by counting a 0.2 ml aliquot dried on a planchet. The radium yield is 75 per cent. The method has been checked by four laboratories and has been in daily use for over six months at the Winchester Laboratory.

A similar carrier-free method was developed for determining thorium-230 but the reliability of this method has not been proven. Development work on thorium procedures is presently underway.

Total alpha count can be determined with the use of either scintillation or gas-flow counters and appropriate scaling equipment.

Equipment Required

The references cited earlier in this report contain detailed information on the equipment required for fluorometric assays of uranium. No special equipment is required for determining uranium in mill effluents.

Section 8 of the Appendix lists the special laboratory equipment and reagents required for the carrier-free radium analyses. The same alpha counting instruments used for counting dust samples are used for counting radium.

The Winchester Laboratory found that new or carefully cleaned polyethylene or Pyrex bottles should be used for collecting samples. One-liter bottles should be used for collecting solution samples as the radium determination requires 500 ml of sample.

Effluent Sampling Program

During the latter part of July, a sampling program of the Monticello tailing pond overflow solution was started for the purpose of radium determinations. Each day two 100-ml grab samples of the tailing pond water are taken simultaneously in new glass bottles. One sample is used for determining uranium, chloride, and pH on a daily basis. The remaining 100 ml is saved for a weekly composite. Each week, a seven-day composite is made up in a new one-liter polyethylene bottle and shipped to the Winchester Laboratory for radium assays. Bimonthly samples of the tailing pond effluent and South Creek, the stream into which effluents are discharged, are taken at four points and analyzed for chemical pollution. The assays for radium on the weekly composites over a period of three months are shown in Table VI. This period includes the time during which processing was converted from two mills (400 tpd acid leach - RIP and 200 tpd alkaline leach - filtration) to a single 300 tpd alkaline leach-RIP mill. After the conversion, radium assays increased. A combined alkaline and acid plant with neutralization of the tailings to pH 7 appeared to result in lower quantities of radium in solution than most other milling processes. The reason for the variation in radium assays from 343 to 2700 dpm/l on the alkaline RIP tails is presently being studied.

For purposes of Part 20 the concentration of radioactive material in water released to unrestricted areas can be averaged over a period not greater than one year. Frequency of samples will depend upon variation in the effluent, type of treatment used, and consistency of removal of radium and/or other isotopes.

Laboratory Effluent Treatment Studies

For the past year the Raw Materials Development Laboratory has been studying radioactive isotopes in mill effluents, developing analytical methods, and investigating various techniques for reducing the concentration of these isotopes which would be

Table VI
Radium Analyses of Monticello Tail Pond Water Samples

<u>Composite Date, 1958</u>			<u>pH</u>	<u>µc/ml x 10⁹</u>	<u>Ra</u> <u>dpm/l</u>
7/28	-	8/3	6.9	244	543
8/4	-	8/10 ^{1/}	7.2	127	282
8/11	-	8/17	10.1	193	428
8/17	-	8/25	10.4	583	1300
8/26	-	8/31	10.2	1040	2310
9/1	-	9/7	10.1	1220	2700
9/8	-	9/14	10.1	433	962
9/14	-	9/21	10.1	417	926
9/23	-	9/29	10.1	550	1219
9/30	-	10/6	10.1	416	924
10/6	-	10/13	10.1	277	615
10/13	-	10/19	10.1	472	1047
10/20	-	10/26	10.1	265	590
10/27	-	11/2	10.1	154	343
11/3	-	11/9	10.2	163	361

^{1/} 8/4: Shut down Acid Rip
8/9: First alkaline RIP tails to pond

applicable and economical for use by uranium mills. A survey of a few mills indicated radium and thorium contents in mill tailings exceeded Part 20 specification by 100 to 2000 times. Uranium content in the tailings was near or below specifications.

Various adsorbents and coprecipitants for radium were evaluated and many variables were investigated including pH, time of contact, particle size of the adsorbent, effect of the presence of slimes, stage addition of adsorbents, and countercurrent treatment. These variables were originally studied mainly on acid tailings. Following conversion of the Monticello Mill to the alkaline process, a sizable portion of the research efforts was devoted to treatment of alkaline tails. Laboratory studies to date have been summarized in a recent Raw Materials Development Laboratory Topical Report.^{1/}

By early October of 1958, the results of these investigations indicated that efficient removal of radium from alkaline tails could be accomplished by a two-stage countercurrent treatment using barium sulfate as the adsorbent (commercial barite was used on the majority of the tests). Contact times of about 100 minutes per stage appeared to be optimum; finely ground barite was more effective; and adjusting the pH of the tail pond water from 10.1 to 8.5 or 9.0 with sulfuric acid resulted in more efficient use of the barite. The presence of very small amounts of slimes resulted in lower decontamination ratios. Solution - barite equilibrium curves indicated as little as 0.25 to 0.50 gram of barite per liter of alkaline tailing solution would lower the radium content to specification levels if efficient countercurrent treatment was employed.

^{1/} DeSesa, M.A., "Interim Report on Investigation into Problem of Radioactive Pollution of Uranium Mill Effluents," WIN-101, December 15, 1958.

Tests Conducted at Monticello

A process survey of the Monticello acid RIP circuit was conducted in early August for the purpose of making a radium balance and obtaining information on the behavior of radium through the circuit.

Samples were collected at 35 points through the mill circuits. Uranium and radium determinations were made on both liquid and solid portions. A 48-hour sampling program was completed during which time samples were taken every two hours. The stream flowing past the mill into which the excess tail pond water is discharged was sampled above, beside, and below the mill. Results of the study are reported in a Raw Materials Development Laboratory Topical Report.^{1/}

In summary, it was found that of the 0.83 mg of radium present in each ton of ore only about 3 per cent was solubilized in the leaching operation. Of this soluble portion, 90 per cent reported to the tailings stream and 10 per cent in the yellow cake. However, after combining the acid and alkaline plant tails and neutralizing to pH 7.0, the pond overflow contained only 2 per cent of the radium originally in solution, or 0.05 per cent of the radium in the ore feed.

A test circuit for treatment of tailings solution with barium sulfate was operated at the Monticello Mill using available acid leach agitators. The purpose of the work was to verify laboratory data by mill scale tests and to determine if the same efficiency of radium removal could be obtained with the use of scaled-up equipment and flowrates.

The flowsheet used, shown in Figure 4, utilized a two-stage barium sulfate treatment of the alkaline tailings pond effluent after it had been neutralized to pH 8.4 with sulfuric acid. An idle agitator was used to settle the barite between stages and after the second contact. No equipment was available for testing countercurrent treatment. There were seven hours of contact between the barite and the liquor in each of the agitators because the equipment was too large for the limited amount of pond water available for the tests. In the laboratory, shorter contact times, in the range of one to two hours, have

^{1/} Whitman, A., Beverly, R. G., "Radium Balance in the Monticello Acid RIP Uranium Mill," WIN-113, December 30, 1958.

1/ Sample points

Figure 4

Flowsheet for Tail Pond Water Treatment Tests

proven to be sufficient.

One gram of barium sulfate per liter was added to each of the contacts by a dry reagent feeder. Sulfuric acid addition was controlled manually as indicated by the use of a pH meter. Acid requirements were 1.9 g/l. Samples of the untreated water, the decanted water after the first stage, and the decanted water after the second stage, were assayed for radium. Results indicated a decontamination in the first stage of treatment of about 95 per cent of the radium in solution. The tail pond water fed to the circuit averaged about 412 dpm/l and after one contact the radium content was lowered to an average value of 22 dpm/l. The second stage of treatment showed little change in radium, and the reason for this effect has not been completely determined as yet.

Before recommendations will be submitted for a flowsheet for treatment of the total tail water released at Monticello, equipment selection must be studied and other reagents, such as commercial coagulants in combination with barite or other adsorbents, must be evaluated. Recent developments on the problems are presented in the Raw Materials Development Laboratory Monthly Progress Reports.

Although laboratory tests have demonstrated that uranium mill tailings, both acid and alkaline, can be decontaminated to a radium level meeting Part 20 specifications, additional test work is in progress to improve the economics of the treatment processes

By efficient metallurgical practice, all existing uranium recovery processes are capable of producing tailings which meet the uranium specifications of Part 20. The decontamination of other isotopes such as Th-230 and Po-210 are being investigated.

PRECAUTIONARY MEASURES

Posting Caution Signs

The pertinent parts of Section 20.203 of Part 20 deal with use of conventional radiation caution signs in areas where the airborne radiation hazard exceeds the established limits and in areas where any appreciable amounts of radioactive materials are stored. In the case of storage of natural uranium the figure for the amount of material present requiring the area to be posted is 100 times the value of 50 μc listed in Appendix C of Part 20. This is equivalent to approximately 16 pounds of uranium. Provisions are also provided for labeling containers in which any appreciable amount of natural uranium is transported, stored, or used. In this case the figure from Part 20 is 10 times 50 μc , or approximately 1.6 pounds of uranium.

Accordingly, the Monticello Mill has placed caution signs at the entrances to the ore crushing buildings and product drying and barreling areas where airborne radioactive materials potentially exceed specifications. The signs were also posted on fences around ore stockpiles and tailings ponds as the quantity of natural uranium stored in these areas exceeds the amount as specified in Appendix C of Part 20. As yet, drums in which concentrate is shipped have not been labeled, pending a decision by the AEC as to the necessity of so doing.

Regulations Regarding Personal Habits and Cleanliness

The Monticello Mill has established Standard Operating Procedures (Section 5 of the Appendix) whereby operators working in the concentrate section of the mill are required to shower prior to leaving the plant. Their work clothes must be left in the plant change room in specially provided areas. Other operators are encouraged to shower, and facilities are furnished along with a place to store their clothing. Plant regulations forbid storage of lunch pails, food, beverages, and smoking materials in certain areas, and eating, drinking, and smoking are not permitted in certain areas.

Clothing in the form of coveralls, rubber gloves, and safety shoes is furnished to operators in the yellow cake area. Maintenance personnel working in this area are also furnished clothing during the periods they are working there. The coveralls are laundered on a daily basis so that each operator has a clean pair each day. The rubber gloves are washed in soap and water at the end of each operating shift.

Procedures for use, care, and cleaning respirators are a part of Standard Operating Procedures (Section 9 of the Appendix) as evaluation of dust surveys and urine samples show the proper use of respirators has been effective in reducing the inhalation in areas of high dust exposure. However, respirators are not considered as protective measures in lieu of permanent and adequate preventative and control measures required for the successful suppression of radioactive dust.

The precautionary measures listed above are a vital part of the personnel education and training program to minimize the opportunity for operating personnel to spread contamination outside of the plant area and to insure a minimum amount of bodily intake of uranium by inhalation or ingestion.

A list of pertinent reference books and bulletins is given in Section 10 of the Appendix.

WIN-114

APPENDIX

O Location of Air Sampling Devices
 HASL - Health & Safety Laboratory Survey
 April 1957
 NLO - National Lead of Ohio Survey
 July 1958
 NL Inc. - National Lead Co., Inc. Survey
 October 1958
 MPC Reference Level: 110 dpm/m³

Appendix Figure 1
Location of Air Sampling Devices and Dust Level
Sample Plant

Appendix Figure 2
Location of Air Sampling Devices and Dust Level
Plant Crusher Building

Appendix Figure 3

Location of Air Sampling Devices and Dust Level
RIP Building

Appendix Figure 4

Location of Air Sampling Devices and Dust Level
Yellow Cake Drying, Barreling, and Storage Area

Appendix Figure 5

NATIONAL LEAD COMPANY, INC.

Job Exposure Evaluation

Survey Period _____

BUILDING _____ PROCESS AREA _____

OPERATOR _____ men/shift _____ shift/day _____ men/day

Operation or Operating Area	Time Per Oper.	Oper. Per Shift	Time Per Shift(T)	Sample No.	Concentration d/m/m ³			TXC
					High	Low	Avg (C)	

$\Sigma (T)$ _____

$\Sigma (Tx C)$ _____

$$\frac{\Sigma (Tx C)}{\Sigma (T)} = \text{_____ d/m/m}^3 = \boxed{\text{_____}} \text{ MPC}$$

Appendix Figure 6

NATIONAL LEAD COMPANY, INC.

Area Exposure Evaluation

Date _____

BUILDING _____ PROCESS AREA _____

OPERATOR _____ men/shift _____ shift/day _____ men/day

Description of Location	Prevailing Conditions	Time Per Sample (T)	Sample No.	Concentration d/m/m ³			T X C
				High	Low	Avg. (C)	

$\Sigma (T)$ _____

$\Sigma (TxC)$ _____

$$\frac{\Sigma (TxC)}{\Sigma (T)} = \text{_____ d/m/m}^3 = \boxed{\text{_____}} \text{ MPC}$$

Section 1

CONDITIONS REQUIRING SPECIAL PRECAUTIONS WHEN DUST SAMPLING

I. Fire and Safety

In an explosive atmosphere only approved-type explosion proof motors shall be used, and the use of spark-emitting devices shall be prohibited.

When sampling stacks or other high places, safety belts shall be used.

The surveyor shall practice good safety techniques and follow all safety rules when collecting samples.

II. Health

1. The surveyor collecting samples in an area of high dust concentration shall provide his own respiratory protective equipment which he shall wear at all times when in the area.
2. The surveyor is expected to comply with all posted rules and regulations pertaining to the area in which he is working.

III. Equipment Care

1. The care of the survey equipment is of utmost importance in survey work. Every precaution shall be taken to protect the equipment and maintain it in proper working condition.
2. The proper care of the equipment would be to prevent damage from dropping the pumps, bumping the rotometers against hard objects or other pumps, and thorough cleaning after the completion of the sampling.
3. The following should be checked to ascertain proper working condition: Motor and bearings are not running hot, the copper tubes are not punctured or flattened, the rotometer is not cracked or broken, the adjusting screw is not bent or damaged, the male fitting for the sample holder is not bent or cracked, the electric cable does not have poor splices or defective male plugs, and that the rotometer registers a maximum flow rate of 30 liters per minute.
4. A check list of the above items should be made by a qualified electrician before the equipment is stored after use and prior to beginning a new survey.

Section 2

STANDARD OPERATING PROCEDURES FOR DUST SUPPRESSION AND CONTROL
OF EXPOSURE TO RADIOACTIVE CONTAMINANTS TO BE FOLLOWED BY
SAMPLE PLANT EMPLOYEES

I. Crusher operators, splitter operators, bucker and sample room operators

1. Buildings and equipment are to be kept clean at all times.
2. Vacuum cleaning is the method to be used to clean dust from floors, walls, ledges, pipes or other places where dust may collect.
3. Compressed air will be used only to clean out equipment between ore lots and when cleaning plugged chutes, etc., while sampling wet, sticky ore.
4. Respirators must be worn at all times when dusty conditions or suspected dusty conditions exist.
5. Lunch pails will be stored in change house where employees will eat their lunches.
6. All employees shall wash their face and hands thoroughly before eating.
7. Water will be applied at all available points when crushing dusty ore.
8. Dust collector will be running at all times while sampling and cleaning up.
9. Every effort shall be made by all employees to prevent dust from entering the body.

II. Truck drivers and loader operators

1. Respirators must be worn at all times while loading or dumping dry dusty ore. Whenever possible, operators should load on the upwind side of operations, thus eliminating dust from blowing on them.
2. Loading areas, coarse ore bin at mill, and the area around the rejects bin will be kept clean at all times.
3. Lunch pails will be stored in change house where employees eating on the job will eat their lunches.
4. All employees will wash their face and hands thoroughly before eating.

Section 3

STANDARD OPERATING PROCEDURES FOR DUST SUPPRESSION AND CONTROL
OF EXPOSURE TO RADIOACTIVE CONTAMINANTS TO BE FOLLOWED BY
CRUSHING SECTION EMPLOYEES

1. Building and equipment are to be kept clean at all times
2. Vacuum cleaning is the method to be used to clean dust from floors, walls, ledges, pipes, and other places where dust may collect. Compressed air will not be used to clean up.
3. Respirators must be worn at all times when dusty conditions or suspected dusty conditions exist.
4. The Rotoclone will be in operation at all times while crushing and cleaning up. Check water level, baffles, mud drag and star feeder before starting.
5. The large doors to the crushing building will be closed at all times.
6. Lunch pails will be stored at Sample Plant change house, where lunch will be eaten.
7. Each employee shall wash his hands and face thoroughly before eating.
8. Every effort shall be made by each employee to prevent dust from entering the body.

Section 4

STANDARD OPERATING PROCEDURE USED IN HANDLING PLANT HEAD, TAIL,
AND CONTROL SAMPLES IN THE PLANT BUCKING ROOM FOR PROTECTION
AGAINST DUST INHALATION AND/OR INGESTION

The pulverizers, crusher, blenders, and splitters are placed under hoods equipped with an exhaust system to remove dust and to prevent its spread into the surrounding area.

I. The sample buckler while preparing any sample involving dried material shall:

1. Wear a respirator at all times.
2. Turn on exhaust fan motor.
3. Adjust dampers in the hood exhaust system so the draft is sufficient to withdraw dust rising from the sample at any given step in the procedure of sample preparation.
4. Wear a respirator when handling head samples in and out of the drying oven, and when crushing samples in the gyratory crusher located in the ball mill area.
5. Keep all sampling equipment clean.
6. Wipe down hoods, ovens, motors, and fixtures with a damp cloth or sponge.
7. Keep floors and windows clean.
8. Keep sample discards cleaned up and rejects emptied on schedule.
9. Use vacuum cleaner as required to keep dust from accumulating.
10. Use air under hoods only and only when absolutely necessary.
11. Wash hands thoroughly with soap and water before eating or smoking and at the end of shift.

II. The product supervisor while working in the yellow cake barreling area shall follow standard operating procedures for dust suppression and control of exposure to radioactive contaminants for that area.

Section 5

STANDARD OPERATING PROCEDURES FOR DUST SUPPRESSION AND CONTROL
OF EXPOSURE TO RADIOACTIVE CONTAMINANTS TO BE FOLLOWED BY
ALL PERSONNEL WORKING IN YELLOW CAKE AREAS

1. Persons working in or visiting the yellow cake areas shall wash their faces and hands thoroughly before smoking, eating, drinking, or chewing.
2. Due to the potential contamination problem, smoking, chewing, eating, or drinking shall be prohibited in the yellow cake areas.
3. Lunch pails, food, drinks, and smoking materials shall not be taken into these yellow cake areas.
4. All skin eruptions, cuts, open wounds, and abrasions shall be covered with a sterile bandage before entering and while working in these areas.
5. Employees working in the yellow cake areas shall shower and change clothes before leaving the plant area. In no case shall clothing worn in the yellow cake areas be worn outside of the plant area.
6. All employees working in the yellow cake areas shall endeavor to prevent yellow cake from entering the body. This will be accomplished by following all rules and practicing good personal hygiene at all times.
7. Employees working in this area shall shower, dry, and submit a urine sample before leaving on off days and immediately upon return to work after off days.
8. After each separate phase of this operation, the operators shall wash off their gloves, remove the gloves, and wash their faces and hands.
9. Maintenance personnel or other persons working or visiting in the yellow cake areas shall wash their shoes or boots thoroughly and remove their coveralls before leaving the area; this applies even though the person is leaving the area temporarily to obtain materials or supplies.
10. Protective clothing such as coveralls shall be kept as clean as possible. In event the coveralls become contaminated, they should be changed for clean ones.

Section 5 (Cont'd)

11. Protective clothing such as coveralls, shoes, boots, or gloves shall not be removed from the yellow cake area.
12. Rubber gloves shall be washed with soap and water before they are removed from the hands at any time.
13. Employees shall keep two pairs of rubber gloves for wear, one pair to be washed thoroughly inside and out at the end of each shift so as to be drying during the following shift.
14. Protective goggles, safety glasses, or face shields shall be worn by employees when on duty in this area or any plant area, the same as applied to all plant employees and visitors in operating areas.
15. Respirators shall be worn at all times when the employees are in the barreling area during barreling operations, when the dryer oven doors are open, and when cleaning up. Respirators shall be turned in weekly for sterilization. This applies whether or not dust is visible in the air.
16. All persons required to use a respirator shall bring the respirator to the first-aid room to be sterilized before leaving the plant for two or more days off and shall obtain a clean respirator before returning to his regular duties after his "off" days.
17. The yellow cake barreling area, press area, and barrel storage area are to be kept free from dust on the equipment, pipes, ledges, and floors at all times.
18. Dust shall be removed with a vacuum cleaner or with a hose and water; never use brooms or blow down dust with compressed air.
19. Splashes and spills shall be cleaned up as soon as possible to prevent contamination of shoes, boots, or clothing.
20. Vent tubes to the scrubber or dust collectors shall be checked and cleaned, if needed, at least once each week and more often if necessary.
21. Doors and entrances to these areas shall be kept closed and chains shall be hooked except when actually in use.
22. All persons (including supervisors) who are visiting the area shall sign in and out in the book provided for this purpose.

Section 6

STANDARD OPERATING PROCEDURE USED IN THE HANDLING OF CONCENTRATE
SAMPLES IN THE YELLOW CAKE PREPARATION ROOM FOR PROTECTION AGAINST
DUST INHALATION AND/OR INGESTION

- I. Yellow cake preparation room employees will comply with the following standard operating procedures:
 1. Coveralls and a respirator will be worn at all times by the yellow cake supervisor when handling yellow cake samples.
 2. Asbestos gloves will be worn while removing hot sample jars from the drying oven.
 3. The exhaust fan shall be in operation and dampers adjusted to exhaust dust from area under hood where sample transfer is being made.
 4. Entire area, including hoods, fixtures, furniture, and equipment, is to be kept clean by using dampened cloth, sponge, or vacuum cleaner only.
 5. Sample jars, lids, and pans are to be cleaned first with a 1:3 hydrochloric acid to water solution followed by thorough washing with fresh water.
 6. Rubber gloves shall be worn while washing and cleaning.
 7. Sample rejects are to be emptied regularly and drawers and cabinets kept free of yellow cake.
 8. Hands and face shall be thoroughly washed with soap and water after finishing preparation of samples, also before smoking or eating and before leaving area at the end of the shift.
- II. The product supervisor while working in the yellow cake barreling area shall follow standard operating procedures for dust suppression and control of exposure to radioactive contaminants for that area.

SECTION 7

FLUOROMETRIC PROCEDURE FOR URANIUM IN URINE

General

To each new urine sample add about 1 per cent by volume of concentrated hydrochloric acid if the sample will not be analyzed within one hour.

The procedure consists of pipetting duplicate 0.2 ml aliquots of a urine sample directly onto a 0.4 gram pellet of 98% NaF - 2% LiF flux.

The majority of the readings fall in the minimum range that can be reliably read on a fluorometer so that extreme care must be taken with the sample. Consistent and reliable results are dependent on clean platinum dishes and the elimination of any possible contamination.

The apparatus and equipment normally used for fluorometric analysis is suitable. Twenty clean dishes should be set aside for use in urine analysis only.

Procedure

1. Prepare a plate of 20 blanks and measure each fluoride disc to make certain dishes are clean. Record the average blank.
2. In a plate of 20 dishes one can use 16 dishes for 8 samples in duplicate and the remaining 4 can be used for the usual 100 mg U_3O_8 standard.
3. Pipette 0.2 ml of urine sample from a clean 0.2-ml pipet into each of two platinum dishes. When a complete set of 8 samples and 4 standards have been pipetted into the dishes, place on a large transite board on a hot plate set at moderate heat.
4. Allow samples to evaporate to dryness.
5. Fuse on the modified Fletcher gas burner, and allow to cool.

6. Adjust fluorometer in the usual manner, and read the fluorescence of the pellets.

7. Record Readings.

8. Calculate results.

Calculations

1. Average the readings from each urine sample.

$$\frac{R_1 + R_2}{2} = R_{\text{avg}}$$

2. Average the four readings from the 100 mγ standards.

$$\frac{S_1 + S_2 + S_3 + S_4}{4} = S_{\text{avg}}$$

3. Subtract the daily blank from each average, and calculate the amount of uranium in each urine sample.

$$\frac{R_{\text{avg}} - \text{Blank}}{S_{\text{avg}} - \text{Blank}} \times 100 = \text{m}\gamma \text{U}_3\text{O}_8 \text{ in } 0.2 \text{ ml of urine}$$

4. Multiply by 0.842 to convert from U₃O₈ to U and by the factor 0.005 to convert mγ U/0.2 ml to mg U/liter of urine.

$$\frac{\text{m}\gamma \text{U}_3\text{O}_8}{0.2 \text{ ml}} (.842) (0.005) = \frac{\text{mg U}}{\text{liter}} \text{ of urine}$$

Cleaning Platinum Dishes

1. Generally, fluxing dishes once or twice with the fluoride flux between runs will clean them sufficiently. However, periodically they should be cleaned in sulfuric and nitric acid as follows:

- (a) Heat dishes in concentrated sulfuric acid for 30 minutes, cool, and rinse dishes thoroughly with distilled water.
- (b) Heat in concentrated nitric acid for 15 minutes, cool, and rinse with water.
- (c) Dry on burner and flux until blanks are at a minimum.

Section 8

THE CARRIER-FREE DETERMINATION OF RADIUM

Apparatus

The counting equipment can be a gas-flow counter with accessory equipment such as proportional counting gas, a lead shield, and a suitable scaler and amplifier. Or a scintillation counter with a crystal sensitive to alpha radiation and a suitable scaler may be used.

Special Reagents

Lead carrier, 0.49 M lead nitrate solution (100 mg Pb⁺⁺ per ml)

6M Ammonium Acetate Solution

25% Sodium Carbonate Solution

Fuming Nitric Acid

1.8 M (15%) Hydrochloric Acid

Dowex 1-X8, 100-200 mesh anion exchange resin

9M (50%) Sulfuric Acid

1M Nitric Acid

Procedure

1. Adjust 500 ml of the sample to pH 1 with concentrated nitric acid and heat to 70°C. While stirring sample with a Teflon-coated magnetic stirring bar, add 5 ml of the lead carrier solution and then add very slowly 60 ml of 9M sulfuric acid. Stir continuously for 1 hour, allow the precipitate to settle, and discard the supernatant solution by careful decantation.

2. Transfer the precipitate to a 50-ml centrifuge tube using 1M sulfuric acid. Centrifuge and discard the aqueous. Add 5 ml of 6M ammonium acetate to dissolve the lead sulfate (all the precipitate may not dissolve since small amounts of alkaline earths may be present). Add 25 ml of 25% sodium carbonate solution and stir intermittently for one hour.

3. Centrifuge, discard the supernatant, and add 3M nitric acid until effervescence ceases. Add water and heat to dissolve the salts. The total volume after addition of acid and water should be approximately 5 to 7 ml. If at this point of the procedure all the salts are not in solution after heating, add 25 ml of 25% sodium carbonate solution; stir for one hour; centrifuge and discard aqueous; and start step 3 again. When the salts are dissolved, place the centrifuge tube in an ice bath; add 30 ml of red fuming nitric acid and stir. Cool, centrifuge, and discard the aqueous phase.

4. Place the centrifuge tube in a water bath and spread the solid material to prevent splattering. Heat to drive off excess acid, add 30 ml of water, and heat if necessary to dissolve the salts. Add 6 ml of concentrated hydrochloric acid dropwise (stirring constantly) to precipitate lead chloride, and cool in an ice bath. At this point, stop the procedure and let the sample sit overnight.

5. The following morning, centrifuge and pass the supernatant solution over a column of 15 ml of Dowex 1-X8, 100-200 mesh anion exchange resin, suspended in 1.8M HCl. Use a flow rate of one ml per minute and collect the effluent in a 150-ml beaker.

The resin bed removes the small amount of lead soluble in 2M HCl while the radium passes through the column.

6. Add 35 ml of water to the lead chloride precipitate, and heat in a water bath with stirring until the solid dissolves. Add 7 ml of concentrated hydrochloric acid dropwise (stirring constantly). Cool in an ice bath; centrifuge; pass the supernatant solution over the same resin bed; and wash the resin bed with 15 ml of 1.8M hydrochloric acid. Collect and combine all resin bed effluents; discard the precipitate.

7. Evaporate the effluent to dryness, but do not bake the sample. Add 10 ml of nitric acid and evaporate to dryness to destroy organic matter from the resin. Wash the beaker with small volumes of 1M nitric acid and transfer to a 10-ml volumetric flask. Evaporate a 0.2-ml aliquot on a stainless steel planchet and count for alpha activity.

Samples should be counted immediately. If this is not possible, the sample should be boiled for 2 to 3 hours before being diluted to final volume.

Calculations

The radium yield is 75 per cent.

$$\text{Counts per minute for sample, cpm} = \frac{\text{total count}}{\text{total time, min.}} - \text{background}$$

$$\text{Disintegrations per minute, dpm} = \text{cpm} \times \frac{100}{\% \text{ geometry}}$$

$$\text{Disintegrations per minute per liter, dpm/l} = \frac{\text{dpm}}{0.75} \times 50 \times 2$$

Section 9

STANDARD OPERATING PROCEDURES REGARDING
THE USE AND CARE OF RESPIRATORY EQUIPMENT

I. Scope and Purpose

This procedure governs the use of respirators, limitations thereof, and the sterilization of such equipment. Respirators are used to minimize the amount of dust entering the respiratory organs of employees. The procedure sets forth regulations for wearing respirators, care of the equipment, and provides for a definite schedule for cleaning and sterilization of respirators to prevent contamination of the equipment or the wearer.

II. Procedure

1. All employees required to work for short periods of time in a known or suspected dusty location shall wear an approved-type respirator.
2. Filters in respirators shall be changed as often as necessary to give maximum filtering effect to the wearer. Anytime visible dust has collected on the filter, the filter shall be changed.
3. Facelets for the respirators shall be changed as often as necessary to insure a clean contact with the face.
4. All respirators shall be brought to the First-Aid Room for sterilization after work each week or before scheduled days off, or more often when considered necessary.
5. In no case shall a respirator that has been stored in a location where it may have become contaminated with dust be used before it is sterilized.
6. The wearing of respirators in designated areas and on specific jobs shall be rigidly enforced by all supervisors.
7. The wearing of respirators in any but the proper position over the nose and mouth shall be considered a violation of the posted safety rules and appropriate action will be taken by management.

Section 9 (Cont'd)

III. Sterilization of Equipment

All respirators and their component parts shall be disassembled and washed thoroughly with warm water and soap and shall be rinsed in warm water before the sterilizing bath. All parts shall remain in the sterilization bath for no less than thirty minutes, after which they shall be thoroughly rinsed and dried.

Before the respirators are reissued they shall be equipped with a fresh filter, and the employee shall install a fresh facelet before using the respirator.

Respirators shall be identified when they are turned in for sterilization so that, so far as possible, the same respirator shall be issued to the employee who wore the respirator originally.

IV. Special Precautions

Respirators shall not be used in place of a self-contained breathing apparatus in locations where toxic or harmful gases are present.

Section 10

REFERENCE BOOKS AND PUBLICATIONS

1. Aspects of River Pollution, Louis Klein, Academic Press 1957
2. Control and Removal of Radioactive Contamination in Laboratories, Handbook 48, U. S. Department of Commerce, National Bureau of Standards, December 15, 1951
3. Control of Radon and Daughters in Uranium Mines and Calculation of Biological Effects, W. M. Gafafer, DSC Tech. Adv., U. S. Department of Health, Education, and Welfare, Public Health Service Publication No. 494
4. Determination of Gross Radioactivity in Water, Silt, and Biological Material, Public Health Service, R. A. Taft Sanitary Engineering Center, Cincinnati, Ohio
5. Health and Safety Considerations for Uranium Fuel Fabrication Facilities, COO-212 (Revised), W. A. Brobst, USAEC Health and Safety Division, Chicago Operations Office, Lemont, Illinois, August 1, 1958
6. Industrial Waste, Willem Rudolff, Reinhold Publishing Co., 1953
7. Living with Radiation, Part I, F. L. Brannigan, USAEC
8. Maximum Permissible Amounts of Radioisotopes in the Human Body and Maximum Permissible Concentrations in Air and Water, Handbook 52, U. S. Department of Commerce, National Bureau of Standards, March 20, 1953
9. Nuclear and Radiochemistry, G. Friedlander, J. W. Kennedy, John Wiley and Sons, Inc., New York, November 1957
10. Permissible Dose from External Sources of Ionizing Radiation, Handbook 59, U. S. Department of Commerce, National Bureau of Standards, September 24, 1954
11. Radiation Hazards in Firefighting, Safety and Fire Protection Branch, USAEC, Technical Bulletin No. 4

Section 10 (Cont'd)

12. Radiation Safety Primer, F. L. Brannigan, 2nd Edition, Supt. of Public Documents, Technical Information Div., Oak Ridge, Tenn.
13. Radiation Safety Primer Instructions Handbook, Safety and Fire Protection Branch, USAEC, Washington, D. C.
14. Radiological Health Handbook, Kinsman, S., Public Health Department, R. A. Taft Sanitary Engineering Center, Cincinnati, Ohio, January, 1957
15. Radiological Monitoring Methods and Instruments, Handbook 51, U. S. Department of Commerce, National Bureau of Standards
16. Regulation of Radiation Exposure by Legislative Means, Handbook 61, U. S. Department of Commerce, National Bureau of Standards, December 9, 1955
17. Some Effects of Ionizing Radiation on Human Beings, edited by E. P. Cronkite, V. P. Bond, C. L. Dunham, USAEC, Naval Medical Research Institute, Bethesda, Md., U. S. Naval Radiological Defense Laboratory, San Francisco, California, Medical Dept., Brookhaven National Laboratories, Upton, California
18. Sourcebook on Atomic Energy, S. Glasstone, second edition, D. Van Nostrand Company, Inc., Princeton, N. J., 1958
19. Standards for Protection Against Radiation, Part 20, Chap. 1 Atomic Energy Commission, Title 10 - Atomic Energy, No. 19, Vol. 22, Federal Register, January 29, 1957
20. Transportation of Radioactive Material, USAEC Division of C. & S., Traffic Management Sect., Washington, D. C.,
21. Water Quality Criteria, State Water Pollution Control Board, Sacramento, California