

FEB 4 1959

LPL:CEM

Docket Nos. 70-36, 40-2134

Mallinckrodt Chemical Works  
St. Louis 7, Missouri

Attention: Mr. W. M. Leaders  
Technical Director

Gentlemen:

This refers to the inspection conducted on the 18th and 19th of August, 1958 of your activities authorized under AEC Special Nuclear Material License No. SM-33 and under Source Material License No. C-2734.

No items of noncompliance were noted as a result of the inspection.

We appreciate the cooperation given the Atomic Energy Commission representatives.

Very truly yours,

DISTRIBUTION

M. M. Mann, INS  
Doc. Rm. (2)  
Formal (2)  
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L&R Reading  
LEL Reading  
C. P. McCallum, LEL

Lynell Johnson  
Chief, Licensing Branch  
Division of Licensing and Regulation

AIR MAIL

E-135

OFFICE ▶	LEL	LEL	LEL			
SURNAME ▶	C. P. McCallum	L. Johnson	L. Johnson			
DATE ▶	2/3/59	2/4/59	2/4/59			

Curtis A. Nelson, Director, Division of  
Inspection, Washington, D. C.

S. R. Sanirle, Manager  
Oak Ridge Operations Office

COMPLIANCE INSPECTION REPORTS

SYMBOL: MI:DCH

Enclosed are two initial inspection reports covering the program conducted by Mallinckrodt Chemical Works at its Hematite, Missouri, plant under Source Material License No. C-273h and Special Nuclear Material License No. SNM-33. Both reports are considered "clear" cases.

Several weak points in the licensee's nuclear safety program were noted and discussed with responsible licensee personnel who stated that measures will be taken where reasonably possible to improve their operation situation. No specific recommendations were made by Division of Inspection personnel.

Additional information concerning the results of radiation surveys and effluent monitoring is considered desirable and this item was discussed with the supervisor of the radiological safety program who stated that future records will reflect a more complete program. This item is not considered sufficient at this time to support a noncompliance citation against the licensee for inadequate surveys.

Company confidential information is contained in Exhibits A and B and in Section VII of the details. This information consists of organization and production data that the licensee prefers not be made available to the general public and competitors.

Original Signed By,  
Leo Dubinski

*for* S. R. Sanirle

Enclosures:  
Inspection Reports as Listed (in dup.)

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studies in connection with the preparation of reactor fuel elements. The research and development work is being carried out by a person licensed by the Commission, within the limits of his license. Conditions required to maintain records of activities are being maintained. The licensee is required to maintain records of activities in accordance with the standards for protection against radiation.

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6. Inspection findings (and items of noncompliance)

The licensee's activities that utilize source material obtained under this license are carried out in the same facilities that are used for a program involving special source material obtained by Hallinckredt under License No. 584-13. Both programs are confined to one plant located in Hazelton, Missouri, which is devoted exclusively to commercial processing and production of uranium compounds and metals. No thorium has been prepared under this license to date but may be obtained at some future date. The licensee's plant is staffed with qualified technical personnel, the majority of whom have had previous experience working with source grade material. A radiological safety program is in effect under the supervision of an experienced person who is both an Industrial Hygienist and a Health Physicist by training. Adequate radiation detection and monitoring devices are available and in operating condition. Personnel monitoring is conducted with the routine use of film badge and bio-assays services supplied by private commercial organizations. In addition, pre-employment and routine physical examinations are given all employees. Routine surveys are made to determine the levels of airborne radioactivity, surface contamination, levels of radiation in process areas and the concentrations of activity in plant effluents. A review of the survey records revealed no significant levels in excess of the permissible tolerances for controlled or uncontrolled areas. The records maintained did indicate that additional surveys and survey information would be desirable in order to give a more complete picture of the plant's radiological safety program. Licensee management stated that this will be done

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7. Date of last previous inspection  None	8. Is "Company Confidential" information contained in this report? Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> (Specify page(s) and paragraph(s)) Section VII of the Details and Exhibits A and B
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DISTRIBUTION:

Division of Inspection  
Washington, D. C. (2)

Copy to EIR 12-12-5 Approved by: Leo Dubinski  
See Paragraphs all Leo Dubinski  
From ECR Oak Ridge Operations Office  
(Operations office)

December 1, 1958  
(Date report prepared)

If additional space is required for any numbered item above, the continuation may be extended to the reverse of this form using foot to head format, leaving sufficient margin at top for binding, identifying each item by number and noting "Continued" on the face of form under appropriate item.



DETAILS

I. GENERAL INFORMATION

9. On August 18 and 19, 1958, a compliance inspection was made of the activities conducted by Mallinckrodt Chemical Works, St. Louis, Missouri, that utilize materials obtained under Source Material License No. C-2734 and Special Nuclear Material License No. SNM-33. The inspected activities are confined to one plant located in Hematite, Missouri.

10. The inspection was conducted by Dr. Marvin M. Mann, Division of Inspection, Washington, D. C., and Leo Dubinski and Donald C. Hubbard, Inspection Division, OROO, who, during the course of the visit, interviewed the following licensee personnel:

Dr. W. M. Leaders - Technical Director of Special  
Metals Division  
Dr. E. D. North - Manager of Hematite Plant  
Mr. J. W. Miller - Supervisor of Industrial Hygiene  
Department

11. The licensee currently holds two licenses that cover the Hematite plant operation--one is for source material and the other is for special nuclear material. The licenses have expanded as follows:

A. License No. C-2734

- (1) Issued September 23, 1955, for 500 pounds of UO<sub>3</sub> to be used in UO<sub>2</sub> reactor fuel element studies.
- (2) Amended November 9, 1955, to allow transfer of UO<sub>2</sub> to other licensees.
- (3) Amended December 30, 1955, to provide for 500 pounds of UO<sub>3</sub> and 1,000 pounds of UF<sub>6</sub> to be used in the preparation of UO<sub>2</sub> for fuel element studies.
- (4) Amended March 9, 1956, to provide for 500 pounds of UO<sub>3</sub> and 4,000 pounds of normal UF<sub>6</sub> for fuel element studies.
- (5) Amended May 24, 1956, to provide for 500 pounds of UO<sub>3</sub> and 5,000 pounds of normal UF<sub>6</sub> for fuel element studies.
- (6) License expired September 1, 1956.

(7) Amended October 9, 1956, to provide for processing and fuel element studies involving up to 500 pounds of UO<sub>3</sub>, 1,000 pounds of UO<sub>2</sub>, and 2,000 pounds of UF<sub>6</sub>.

(8) Amended March 19, 1957, to provide for 3,100 pounds of normal uranium metal and compounds for fuel element studies, processing, and resale.

(9) Amended April 25, 1957, to provide for 500 pounds of UO<sub>3</sub>, 1,000 pounds of UO<sub>2</sub>, and 2,000 pounds of UF<sub>6</sub>.

- (13) Amended November 22, 1957, to provide for unlimited amounts of normal uranium metal and compounds to be used for processing, fuel element studies, and resale.
- (14) Amended April 14, 1958, to provide for unlimited amounts of normal uranium and thorium metals and compounds for processing, fuel element studies, research and development activities, and resale.

B. License No. SNM-33

- (1) Issued July 18, 1956, to provide for unlimited amounts of uranium enriched up to 7.5% to be converted to UO<sub>2</sub> using procedures set forth in the licensee's letter dated May 14, 1956. The procedures included design information pertaining to plant facilities, shipping containers, criticality control, process equipment; radiological safety procedures; and detailed process procedures.
- (2) Amended August 8, 1956, to provide for unlimited amounts of uranium enriched up to 7.5% and 1.1 kg. of 20% enriched uranium. Use is according to additional procedures dated June 18, 1956.
- (3) Amended August 24, 1956, to provide for unlimited amounts of enriched uranium to be used according to additional procedures dated August 9 and August 21, 1956. The procedures involve essentially the same process as described in earlier correspondence but with some minor equipment and chemical flow-sheet changes.
- (4) Amended February 19, 1957, to allow the preparation of uranium oxide and uranyl sulfate from UF<sub>6</sub> according to procedures dated January 21, 1957. The procedures describe in detail the process, including criticality considerations, for enrichments ranging from 20% to full enrichment.
- (5) Amended April 22, 1957, to include procedures dated March 5, 1957, which consisted of changes in transportation arrangements and design of shipping containers.
- (6) Amended October 28, 1957, to include procedures dated October 21, 1957, that set forth a mode of operation used for the conversion of UF<sub>6</sub> to UO<sub>2</sub> at assays between 3 and 5% enrichment.
- (7) Amended March 3, 1958, to include the preparation of uranium metals and compounds, blending operations and waste recovery as described in three procedure supplements dated February 4, 1958. The procedures cover equipment and techniques used to produce uranium metal of any enrichment and blended batches of UO<sub>2</sub> up to 5% enrichment. Also described were two facilities used for solvent extraction recovery of uranium from barren material. One facility is designed to handle barren to 5% enrichment while the other is designed to process barren containing from 5% to 20% enrichment.
- (8) Amended April 2, 1958, to include procedures for the preparation and waste disposal of uranium metal.

processes for producing uranyl nitrate and uranyl fluoride (crystals and solution), and shipping containers for the  $UO_2F_2$  and  $UO_2(NO_3)_2$ .

12. In addition to the source material activities conducted at the Hematite plant by the Special Metals Division, the licensee also carries on two additional source material programs. One program, conducted by the Sales Group under Source Material License No. D-217, is a nonprocessing type activity limited to 900 pounds of source material for resale and use as an analytical reagent. The other program involves the processing of euxenite ore for the Government Services Administration under that agency's Source Material License No. D-195. This process is conducted by the Operations Group in one of the licensee's buildings located in St. Louis. Uranium obtained from the ore is sold to the Commission's Weldon Spring, Missouri, Feed Materials Plant, while the thorium residues are stored at the Army's Granite City Engineer Depot in Granite City, Illinois.
13. One additional special nuclear material license has been issued to the licensee for research purposes to develop a process for producing  $UF_4$  by a direct conversion from  $UF_6$ . All development work for this process will be done at the Hematite plant if the research efforts are pursued.
14. The details of this report cover only the activities conducted under License Nos. C-2734 and SNM-33.

## II. LICENSEE BACKGROUND

15. The Mallinckrodt Chemical Works was one of the first companies to commercially produce uranium and uranium compounds and has been continuously engaged in the field since 1942. The company is a prime contractor of the U. S. Atomic Energy Commission and currently operates the Commission's uranium refinery plant at Weldon Spring, Missouri, and formerly operated a similar plant on Destrehan Street in St. Louis, Missouri.

## III. ORGANIZATION

16. The licensee is a stockholder company organized so as to have seven major operating groups as shown in Exhibit A. The Uranium Division of the company is the group responsible for the contract operation of Commission-owned facilities in Weldon Spring in which refined uranium oxides, uranium tetrafluoride and uranium metal are produced.

Following the 1954 amendment to the Atomic Energy Act, which was designed to encourage private business participation in the atomic energy field, the licensee created the Special Metals Division to further the company's private commercial efforts.

17. The Special Metals Division was formed on January 1, 1956, and placed under the management of Mr. Frederick M. Belmore who holds a B. S. degree in chemistry and a B.S.E. degree in chemical engineering. Mr. Belmore was employed in various capacities by the Manhattan Engineer District and the Atomic Energy Commission from 1943 until 1954. The last Commission position held by Mr. Belmore was that of Deputy Director of the New York Operations Office. In addition to being Manager of the Special Metals Division, Mr. Belmore is also Special Assistant to the President of the company, Mr. Fistere, and the Special Metals Division reports directly to Mr. Fistere. Dr. W. M. Lister, Technical Director of the Division, is also the Technical Assistant to Mr. Fistere. The Hematite plant operation is under the management of Mr. Lister.

assisted by the Plant Manager, Dr. E. D. North, and Dr. G. W. Thompkins who directs research and development efforts at the Hematite plant (see Exhibit B).

18. A. The plant operating force consists of approximately 25 persons working under the supervision of the Plant Manager, Dr. North. Reporting to Dr. North directly are two Process Engineers, who act in staff capacity, and a General Foreman. Other supervision includes the three Assistant Foremen, one to a shift, who report to the General Foreman. In the absence of the Plant Manager and the General Foreman, the Shift Foreman is the senior and responsible supervisor of operations. In addition to the operating personnel there are several other persons such as mechanics, porters, and guards who also report to Dr. North.

The Process Engineers are both graduate chemical engineers who normally work day shift but often work around the clock if a situation arises that requires non-routine hours. Research and development people have on occasions in the past acted as relief for the Process Engineers. The Process Engineers' major responsibilities are to check on personnel to determine if written procedures and standards are being complied with and to review each morning all process reports that were completed on the night shifts. Both men have read Los Alamos and Oak Ridge criticality data reports and have copies available for their use but they are not regarded as criticality experts.

No technical training is required for any of the foremen or operators and generally speaking the foremen are old-time employees who have come up through the chemical operator ranks. Operators start as trainees and progress through B and A operator grades, then utility operator with the next promotion being to that of Assistant Foreman.

- B. The plant has a small analytical laboratory, employing five persons, that is maintained and operated by the operating organization. The research and development laboratory, a separate group, is staffed by several technical persons and technicians from the Research and Development organization of the Special Metals Division who are not responsible to the Plant Manager but report instead to Dr. Shearer through Dr. Thompkins.
- C. It appears that operating personnel have been selected with care and that all supervisors and senior operators have some experience in normal uranium processing. Experience in processing of enriched uranium prior to initiation of the Hematite operation, however, is confined to Dr. Leaders, Technical Assistant to the Manager of the Special Metals Division.

#### IV. FACILITIES

19. The Hematite facility is located on a 15-acre tract of land approximately 10 miles north of St. Louis, Missouri, and is bounded on the north by a public road, on the east by Little Creek, and on the east and west by other private property. The plant is centrally located on the 15-acre tract.

On the east side of the main building is the incoming storage and blending building while on the west side is the outgoing storage building. The entire plant area is surrounded by a fence and the gate is manned by guards twenty-four hours a day. A new building duplicating the main building in size is now under construction outside of the fence. When completed, the building will be occupied by both the research and development and operating groups. The fence will be enlarged in the future to include the new building.

20. The main building consists of three separate processing areas that are separated so that materials of various enrichments may be manufactured simultaneously.
  - A. Area number one is designed to handle materials of high enrichment, that is 20% U-235 and higher. The room houses a general products line primarily used for high-fired uranium dioxide for cermet type fuel elements and a process line for the manufacture of metal. Operating in conjunction with the high assay production unit are two auxiliary areas, one containing special equipment used for the solvent extraction of highly enriched uranium from scrap material while the other, a soluble products area, is used for the preparation of crystals or solutions of uranyl nitrate, uranyl sulfate, and uranyl fluoride.
  - B. The second area is for large scale production of low enrichment materials that range up to 5% enrichment in U-235. The main product is ceramic grade uranium dioxide. The equipment in this area, like that in area number one, is also housed in special hoods for dust control and consists of standard chemical plant equipment--tanks, pumps, filter processes, resistance dryers, resistance furnace, etc.
  - C. The third processing area is actually divided into three sections. One section contains equipment for solvent extraction of low enrichment uranium from scrap, while another is for the manufacture of middle enrichment (5 - 20% U-235) uranium compounds. The third section contains the research and development activities and a pilot plant for pressing and firing ceramic pellets of  $UO_2$ . The pelleting line equipment can be used for all assays of uranium and while said to have been initially installed primarily for quality control purposes in manufacturing ceramic grade oxide, it is also used for the production of high density  $UO_2$  pellets for direct loading into fuel elements.
21. The incoming material storage vault is a one-story reinforced concrete building (~30' x 15') equipped with a concrete floor and is located approximately 50 feet to the east of the main building. The room is equipped with chains and brackets that are fastened to the wall and used to secure shipping containers. All shipping containers containing uranium of the same degree of enrichment are chained together. Vault responsibility lies primarily with the process engineers who are assisted by the foremen.
22. The blending building is a single room contained in a concrete block building (20' x 50') located directly south of and adjacent to the incoming storage vault. Blending equipment is contained within a dust control hood and consists of ten 15-gallon open-top drums mounted on a rack in back of a roller conveyor. The rack is separated from adjacent drums by a one-foot gap between drums.

The south end of the room is used for the storage of containers which are designed so as to give a minimum two foot spacing between adjacent containers.

23. The outgoing storage vault is a small (~20' x 10') reinforced concrete building located approximately 50 feet to the west of the main building. The room is equipped with racks and chains attached to the wall that are used to keep containers separate prior to shipment.

#### V. RADIOLOGICAL SAFETY PROGRAM

24. The over-all radiological safety program of the Hematite plant is under the direct supervision of J. W. Miller, Supervisor of the Industrial Hygiene Department, who reports to Mr. J. G. Moore, Vice-President in charge of Operations. Mr. Miller holds a B.S. degree in chemistry and has had several years' experience in the licensee's Uranium Division's Health Division covering similar normal uranium operations. He is by training both a Health Physicist and Industrial Hygienist and is responsible for all of the licensee's commercial operations, including the Hematite plant. He is assisted by two technicians. Control of factors affecting criticality are not part of the radiological safety program but are handled separately by Dr. Leaders and Dr. North with Dr. Leaders having the prime responsibility.
25. Personnel monitoring is conducted by routine use of film badges, bio-assays, and physical examinations. Film badges are obtained from St. John X-Ray Laboratory in Califon, New Jersey, and are distributed to personnel on a frequency determined by individual job assignment. Permanent operating personnel at the Hematite plant are monitored on a weekly basis while rotating personnel are monitored monthly. Complete individual exposure records and summaries are maintained on all employees and were reviewed. The average exposure of Hematite plant personnel during the past year, as indicated by the records, was 80 mrad due to beta radiation and 36 mrad due to gamma radiation. The maximum single six months accumulative exposure recorded as due to beta was 2525 mrad while the maximum due to gamma was 380 mrad. No single weekly exposure in excess of the permissible weekly exposure was noted. Film badges are worn by all persons entering the Hematite plant and are distributed and collected by the guard at the door.

Clinical and radiological urinalyses are made routinely with the frequencies determined by individual job assignment. The minimum is one analysis per year while the maximum number of routine analysis per person is four per year. The radiological urinalyses are performed by a commercial organization, Nuclear Service and Engineering Corporation, located in Pittsburgh, Pennsylvania. This service was formerly supplied by the Tracerlab Corporation. Results are reported in disintegrations per minute per liter (~24 hour sample) with  $46 \text{ d/m/l}$  established as tolerance. The maximum single exposure, as indicated by the licensee's records, was  $37.4 \text{ d/m/l}$ , while the average during the past year was of the order of  $5 \text{ d/m/l}$ . Pre-employment physicals are given all employees.

Protective clothing, including shoes, are provided and maintained by the company in their own laundry facility. A dedicated change room is used by employees to prevent street clothing from being carried into areas where there is a possibility of contamination.

26. The licensee's airborne radioactivity control program operates on the philosophy that a correctly designed installation will control the material in such a manner that the material does not become a health hazard to the employee who operates the installation. Thus, maximum attention is given to adequate engineering design and to the development of standard operating procedures which will provide acceptable control. Dust studies (general and breathing zone) of individual jobs are made to determine an operator's exposure to airborne material while carrying out his normal duties. Based upon the dust studies, changes are made to achieve the lowest practical exposure. Concentrations greater than the plant's maximum permissible concentration ( $70 \text{ } \mu\text{d}/\text{m}^3$ ) during some operations may be permitted provided the weighted average for the daily job does not exceed one M.P.C. In general, design and operation are aimed toward an upper limit of  $1/10$  M.P.C. Once a job has been evaluated, it is assumed that the exposure of each operator on that job will be approximately the same. The exposure value for that operation is then charged to each person who performs the operation. A complete job history is maintained on each employee and a re-study of each operation is made on a periodic basis to establish an up-to-date exposure value. High urinalysis results and film badge reports also require a re-evaluation of an operation.

Air samples have been taken on the roof of the main building with the maximum results noted being  $1 \times 10^{-12} \text{ } \mu\text{c}/\text{ml}$  of air. Samples have not in the past been taken outside of the fence or at the outer edge of the property line, but plans are being developed to do so in the near future.

All dusty operations are enclosed in hoods equipped with double filters, and, in addition, all personnel are provided with face masks which are worn around the neck when working with a dust generating process even though the process is enclosed in a hood.

Complete air surveys are made of the entire plant at least twice a year and some sampling is done in the plant at least one week out of every month; however, no full-time Health Physicist is assigned to the Hematite plant.

27. Area monitoring of the entire plant, using survey meters, is done at least four times a year formally and more often informally. The smear technique for detecting contamination is not used. The results of the four formal surveys are recorded on floor plans which are filed in Mr. Miller's office. The records available were reviewed with no significant radiation levels noted; however, it was pointed out to Mr. Miller that both his area monitoring and air sampling survey records were considered to be close to marginal since they did not present a complete picture of the Hematite operation. He stated that the program would be expanded in the future in order to fill in the gaps noted in his present records. It was also pointed out that instrument surveys are not always adequate for revealing low level alpha contamination and that since he had counting equipment available he might want to evaluate the use of smears for that purpose.

28. Instrumentation for the radiological safety program consists of:

2 - Technical Associates Model No. 3 June 1954  
3 - Victoreen Physics Model 3870 Survey Meter  
3 - Victoreen Model 3870 Survey Meter

29. Most of the contaminated liquid waste generated by the licensee's activities consists of ammonium diuranate. These solutions are currently treated with lime to precipitate the uranium and fluoride values then the slurry is brought to the boiling point to release the ammonia while the precipitate is filtered to remove the calcium fluoride and uranium contents. The filter cake is stored for future processing while the filtrate which is essentially pure water is released to the process waste sewer line. The ammonium fluoride solution analyses have shown the maximum uranium contents to be 50 parts per million with the average value between 5 and 25 parts per million. Ninety-nine per cent of the ammonium fluoride liquors are produced in the section of the plant that handles up to a maximum of 5% enriched uranium. Since the uranium contents are so low the licensee in the near future proposes to stop the lime treatment and transport the untreated ammonium fluoride by truck to its main plant in St. Louis where the material will be used in another process. This procedure has been approved by the Division of Licensing and Regulation.

Liquid effluent monitoring of the Hematite plant discharge is conducted at several locations on a non-routine basis using a grab sample technique. All process wastes are discharged due west from the main building through a sewer that empties into a stream running south through the licensee's property. From there it flows several hundred feet then discharges into the Joachim Creek which is not on licensee's property. Samples have been obtained from the process sewer, from process filtrate, in the stream, and at several points in Joachim Creek, both below and above the entry point of the licensee's stream. Effluent sampling records were reviewed and the highest concentration noted was  $6.03 \times 10^{-6}$   $\mu\text{c}/\text{ml}$  of alpha activity which was detected in the filtrate. The highest concentration reported off the licensee's property was detected in Joachim Creek just below the licensee's point of entry. This was detected on July 27, 1957, and showed a concentration of  $1.84 \times 10^{-7}$   $\mu\text{c}/\text{ml}$  of alpha activity.

It was pointed out to Mr. Miller and Dr. Leaders that with the company's increase in production during the past year it would appear that additional and more recent effluent information obtained on a more routine basis would be desirable for their records. Mr. Miller stated that there were some additional sample results recorded but that he was unable to locate them during the visit. He also stated that the stream on the company's property will be monitored with a continuous sampler in the near future. No figures were available as to the average volume of the plant's liquid effluent or the average flow of Joachim Creek.

30. Shipping containers are monitored by a Health Physicist to assure compliance with ICC regulations and all containers are posted externally with ICC type labels.
31. Regulation type posting is in use throughout the plant including each individual container of material. A color coding system has been combined with container posting which identifies the contents of the material in the containers.
32. As a result of the Oak Ridge (Y-12) criticality incident the licensee is now in the process of obtaining a radiation detection and alarm system that will be installed at several locations throughout the plant. A system designed by the Radiation Control Laboratory is currently being installed.

33. Radiological safety education and instruction of personnel has been conducted on a limited scale since most of the operating personnel now employed have worked either in the company's Uranium Division or the Euxenite Plant. Several lectures are said to have been given by Dr. North on a non-routine basis that were concerned with housekeeping items observed by him during tours through the plant. Written instructions consist of one four-page memorandum entitled "Health Procedures - Hematite Plant" written by Mr. Miller and distributed to top supervision. Copies were observed posted on bulletin boards located at several prominent points within the main building.

VI. PROCESSES

34. The main process of the plant is one whereby uranium dioxide ( $UO_2$ ) is produced from normal and enriched grade hexafluoride ( $UF_6$ ) through an intermediate diuranate (ADU) step. Although the scale of equipment is different depending on whether high or low enriched material is being processed, the basic flow sheets for the production of ADU type  $UO_2$  are identical. Basically, the process consists of the hydrolysis of  $UF_6$  with a dilute ammonia solution to give a precipitate of ammonium diuranate which is filtered, washed, and dried. The dried powder is pyrohydrolyzed with steam and at the same time the ADU is converted to black oxide ( $U_3O_8$ ), which provides an intermediate that is used for preparing other uranium compounds. The  $U_3O_8$  is then reduced to  $UO_2$  with hydrogen or cracked ammonia. If the  $UO_2$  is to be pressed into pellets for use in fuel elements it is blended, pelleted, and sintered at high temperatures.  $UO_2$  high fired crystals and ceramic grade  $UO_2$  may also be produced following the blending step or the  $UO_2$  may be converted to green salt ( $UF_4$ ) for use in preparing uranium metal. Although Mallinckrodt is licensed for processing thorium, none has been obtained to date.

Process scrap, including such items as filter bags, clean-up scrap, rejected pellets, destructive test samples, and analytical scrap, is dissolved in acid; following this the uranium values are extracted in solvent then put in a purified uranyl nitrate solution which can be converted to ADU. Scrap from the processes is recovered from the hoods, filters, and equipment and returned to the customer.

VII. MATERIAL ACCOUNTABILITY

35. Adequate material accountability procedures are in effect. Source and special nuclear materials are received in cylinders as  $UF_6$  which are shipped to the Hematite plant by private commercial truck lines. When the trucks arrive, the cylinders are unloaded, weighed, and stored under the supervision of the Foreman who enters the number and contents of each container in a log book kept in the Foreman's office. Cylinders are removed only when they are used in the process and are immediately accounted for. The balance of the cylinders is maintained in the Foreman's office.

shipments from the Hematite plant and he carefully checks all users to make certain that they are either valid licensees or else are exempt from licensing.

36. The amount of source and enriched grade uranium processed and shipped by the licensee each year is quite large. For example, during 1957 the following amounts of material were handled:

A. Source Material

UF <sub>6</sub> received	11,901.98 lbs.
UF <sub>6</sub> used	11,038.75 lbs.
UF <sub>6</sub> on hand (1-1-58)	863.23 lbs.
UO <sub>2</sub> purchased	8,306.76 lbs.
UO <sub>2</sub> shipped	1,247.88 lbs.
UO <sub>2</sub> on hand (1-1-58)	
Research Department	349.67 lbs.
Warehouse	6,634.90 lbs.
Residues (U content unknown)	156.92 lbs.

The licensee has found it uneconomical to completely empty the source grade UF<sub>6</sub> cylinders, and, as a result, each cylinder returned to the Commission's Paducah, Kentucky, plant contains approximately 1.5 pounds of UF<sub>6</sub>. In arriving at an over-all material balance in the above figures the licensee charged themselves with the total weight of UF<sub>6</sub> recorded in the original transfer papers though it is estimated that the returned UF<sub>6</sub> is approximately 1/4 of 1% of the amount received.

B. Special Nuclear Material

(1) Received

UF <sub>6</sub> (2.3%)	1877.66 lbs.
UF <sub>6</sub> (2.7%)	6936.68 lbs.
UF <sub>6</sub> (1.5%)	4721.604 lbs.
UF <sub>6</sub> (5.4%)	16.24 lbs.
UF <sub>6</sub> (6.8%)	11.257 lbs.
UF <sub>6</sub> (20%)	308.216 lbs.
UF <sub>6</sub> (90%)	149.950 lbs.
UF <sub>6</sub> (NS-40)	362.359 lbs.

(2) Shipped

UO <sub>2</sub> (2.3%)	1534.0 lbs.
UO <sub>2</sub> (2.7%)	6722.63 lbs.
UO <sub>2</sub> (1.5%)	2589.68 lbs.
UO <sub>2</sub> (5.4%)	16.24 lbs.
UO <sub>2</sub> (6.8%)	11.034 lbs.
UO <sub>2</sub> (20%)	173.784 lbs.
UO <sub>2</sub> (90%)	57.277 lbs.

(3) On Hand

(a) In Research Laboratory (1-1-58)

UO <sub>2</sub> (2.3%)	12.15 lbs.
UO <sub>2</sub> (2.7%)	18.84 lbs.
UO <sub>2</sub> (1.5%)	4.295 lbs.

(b) In Process (1-1-58)

UO <sub>2</sub> (20%)	1.446 lbs.
UO <sub>2</sub> (90%)	7.813 lbs.

(c) In Residues (1-1-58)

UO <sub>2</sub> (2.3%)	29.74 lbs.
UO <sub>2</sub> (2.7%)	132.87 lbs.
UO <sub>2</sub> (1.5%)	90.94 lbs.
UO <sub>2</sub> (5.4%)	.007 lbs.
UO <sub>2</sub> (6.8%)	.05 lbs.
UO <sub>2</sub> (20%)	.54 lbs.
UO <sub>2</sub> (NS-40)	15.89 lbs.

(d) In Storage Vault (1-1-58)

UO <sub>2</sub> (1.5%)	2002.264 lbs.
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The above figures indicate the scope and adequacy of accountability procedures.

VIII. CRITICALITY CONTROL

37. The development of a new process prior to putting it into full scale production is carried out in the early stages by the Research and Development Group which devises the chemical flow sheet to be used. The chemical flow sheet is reviewed jointly by the Technical Director, the Production Group, and the Research and Development Group. Following this review the Technical Director, with consultation from Oak Ridge and Rocky Flats personnel, reviews the proposed process from a criticality viewpoint. After the criticality study has been completed, equipment is obtained or designed in accordance with the findings of the Technical Director. The process and equipment are then written up in the form of a license application and submitted to the Division of Licensing and Regulation for approval. When approval has been obtained, the equipment is installed and several test runs are made using source grade material. During these tests the wrinkles are ironed out and an operator's process flow sheet is developed and written up. The entire process, the equipment, test results, and flow sheets are then again reviewed by the Production Group, the Technical Director, and the Research and Development Group. If the review is favorable, the form of the operator's process flow sheet is prepared and the sheets are printed up for use in the production process. When the sheets have been received, source grade material is injected into the system by a person who is followed closely by the three reviewing groups. The test runs are made and the three groups are satisfied that the process is turned over to production personnel. No further review is made of the process after this point.

the procedures and standards were undergoing review and would be revised to conform with experience gained during the past months.

38. Once a process is turned over to the Production Group, the foremen and process engineers assume primary responsibility to Dr. North for monitoring the process to ensure that the operations are carried out according to the developed procedures. These men are aware of the criticality limitations for each step of the process and for each piece of equipment and are responsible for seeing that the operators do not exceed them. The foremen and engineers are continuously circulating in the areas during the production and handling of source and enriched grades of uranium. Operator process flow sheets are reviewed at least once daily to ascertain that procedures are being followed. All operators are said to have been instructed to contact the foremen or engineers if they are uncertain about any phase of their work or if there appears to be an unusual situation arising.
39. Material is received and shipped in approved "always safe" containers that utilize the volume limitation and physical separation principles to prevent the accidental accumulation of a critical mass. These "bird-cage" containers are also used for storing the raw material as well as the finished product. Movement of material within the building is normally done by hand and is usually carried out within dustproof hoods that are physically divided. Foremen are said to control and direct all movements of material in order to prevent more than one container from being in motion in one area at the same time. Equipment in each of the process lines has been so dimensioned as to be "always safe" for the intended enrichment of material. It appears that thorough and conscientious use of experienced consultants has been made in an effort to ensure adequacy of design. All vessels, retorts, trays, and pipes are so sized as to enjoy a factor of safety of approximately two or greater, the factor being based on Oak Ridge and Los Alamos data.
40. It appears that the plant equipment has been carefully designed to minimize the probability of accidental accumulation of critical masses of fissionable materials. There are, however, a number of points in the processes and in the laboratory activities wherein mistakes could occur and critical masses could be assembled. The ones noted are as follows:
  - A. The raw material for the several processes is  $UF_6$ . This material is received from Oak Ridge in "always safe" containers for the material involved. The selection of appropriate containers from storage and the introduction of material to the process line usually depends upon one person. Records of containers and their contents are kept in the Manager's office and the Manager's written approval is required before removal of a container from storage, but the actual removal and placement of material into the process depends upon correct action by this one person. It does not appear that a procedural or analytical check exists for this operation; therefore, the introduction of highly enriched material into the processes for intermediate enrichments is possible.
  - B. At several points in the processes, "always safe" trays are carried by hand from one piece of equipment to another, and, in some cases, trays of enriched material are stored temporarily on tables. Safety in these operations is clearly dependent on proper actions by individuals.

- C. It was observed that in connection with research and development activities, bottles containing uranium solutions are stored temporarily on shelves without physical limitation on geometry and quantity. At the time of inspection, each of some three bottles grouped on a shelf were observed to contain more than 100 grams of Uranium 235 in solutions of greater than 20% enrichment.

After the operations had been reviewed, a conference was held with Dr. North, Plant Manager; Dr. Leaders, Technical Director; and Mr. Miller, Industrial Hygiene Supervisor. The specific items noted above were discussed in some detail and the inspection representatives pointed out that by and large the operation is considered as being well designed and responsibly supervised. However, it was noted that, in the opinion of the Division of Inspection, reliance upon individuals at certain points in the process presents a potential hazard and that improvement in operational safety could possibly be realized by further development of procedure and procedural checks. The subject of divided responsibility in regard to research and development activities was mentioned, particularly since it had been observed as noted in section C above that certain questionable procedures were being followed by research personnel. It is the understanding of the Division of Inspection that Mallinckrodt management is taking steps to improve the situation.

#### IX. SUMMARY

41. The use of source and special nuclear materials in the activities conducted by Mallinckrodt Chemical Works at the company's Hematite, Missouri, plant are in accordance with the license conditions, license applications, and license application supplements.
42. Plant supervision appears to be composed of experienced and qualified personnel who are interested in conducting safe nuclear and radiological programs in conjunction with the licensee's activities. The radiological safety program under Mr. Miller's direction is functioning effectively but additional survey and effluent monitoring information appears to be desirable. A more complete program covering these points is said to be planned for the future. The nuclear safety program under the direction of Dr. Leaders, while adequate from the equipment design point of view, does involve activities at several points in which there is reliance upon individuals to carry out procedures. Any reduction in the procedures which would limit reliance on individuals would further improve the over-all safety of the operation. Management intends to continuously review these matters and make improvements where reasonably possible.
43. It appears that Mallinckrodt management, immediately related to the Hematite operation, understands fully the questions raised by the Division of Inspection and intends that the operation shall be conducted safely.
44. No specific recommendations are made at this time.

UNANIM DIVISION  
Vice President  
Mr. H. E. Taylor

SPECIAL AGENT  
Mr. F. M. [unclear]  
Spec. Agent in Charge

OPERATIONS  
Vice President  
Mr. J. C. Moore

Dr. V. Wallingford  
Senior Director  
Chem. Res. Dept. [unclear]

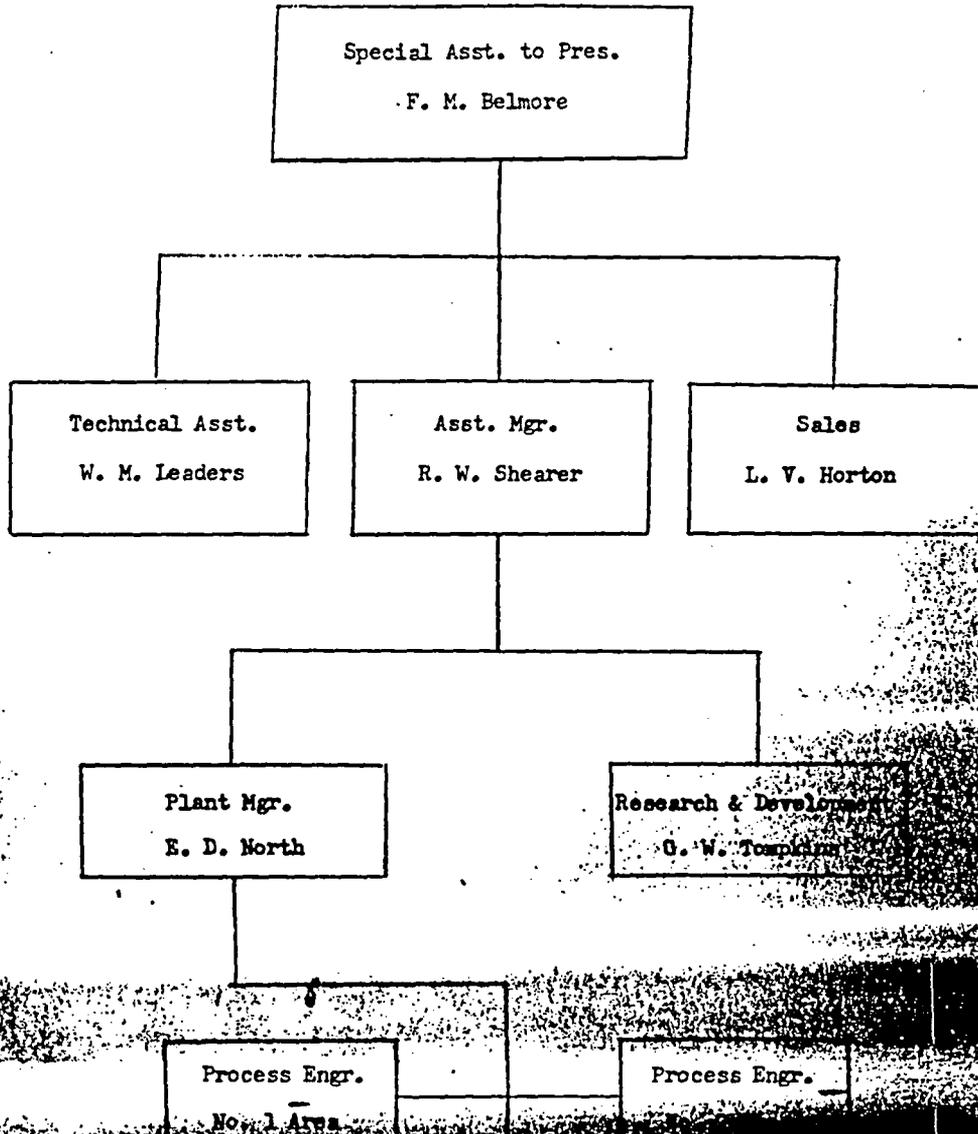
Mr. H. V. [unclear]  
Senior Vice President

ORGANIZATION MANUAL  
UNANIM DIVISION  
SUBJECT

MALLINCKRODT CHEMICAL WORKS

Organization Chart  
Special Metals Division  
of  
Mallinckrodt Chemical Works

August 1958



COMPLIANCE INSPECTION REPORT

1. Name and address of licensee  Mallinckrodt Chemical Works Second & Mallinckrodt Streets St. Louis 7, Missouri	2. Date of inspection  August 18 and 19, 1958
	3. Type of inspection <u>Initial</u>
	4. 10 CFR Part(s) applicable  20 and 40

5. License number(s), issue and expiration dates, scope and conditions (including amendments)

<u>License No.</u>	<u>Date</u>	<u>Expiration</u>	<u>Scope and Conditions</u>
MM-33, as amended	4-28-58	7-1-61	Scope: Uranium enriched in the U-235 isotope, limited to that which may be used in accordance with the procedures specified in Item 8 below, for use in preparation of uranium metal compounds, blending, pelleting, waste recovery, and fuel preparation to the extent described in and in accordance with the procedures in licensee's applications of May 15, June 18, and August 9 and 21, 1956; Jan. 21, March 5, Oct. 21, 1957; and three applications dated Feb. 4, March 14 and April 7, 1958; and waste disposal in accordance with the procedures in the licensee's April 7, 1958 application. Conditions: #10 - Authorized place of use: The licensee's plant near Hazelton, Missouri, as described in the licensee's application. #11 - All transfers of uranium enriched in the U-235 isotope are to be made in accordance with the procedures approved by the Commission.

6. Inspection findings (and items of noncompliance)

Licensee activities utilizing special nuclear material obtained under the license are carried out in the same facilities used for a program involving source material processed under License No. G-2734 also issued to Mallinckrodt Chemical Works. Both activities are conducted in one plant located in Hazelton, Missouri, which is devoted exclusively to commercial processing and production of uranium compounds and metal. The licensee plant is staffed with qualified technical personnel, the majority of whom have had previous experience working with source grade material. A comprehensive safety program is in effect under the supervision of an experienced person who is both an Industrial Hygienist and a Health Physicist by training. Instruments and monitoring devices are available and in operation. The licensee's monitoring is conducted with the routine use of film badge and pocket dosimeters supplied by private commercial organizations. The licensee's monitoring program is comprehensive and gives a continuous record of radiation levels in the plant areas and the concentrations of activity in the plant's effluents. The licensee's survey records revealed no significant levels in excess of the permitted limits in controlled or uncontrolled areas. The records maintained indicated that additional data and survey information would be desirable in order to give a more complete picture of the licensee's radiation protection program.

7. Date of last previous inspection: \_\_\_\_\_

8. If "Completely Confidential" information is contained in this report, check "Yes" in the box below. (Specify page(s) and paragraph(s) if not all of report is confidential.)

Yes <input type="checkbox"/>	No <input type="checkbox"/>
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DETAILS

I. GENERAL INFORMATION

9. On August 18 and 19, 1958, a compliance inspection was made of the activities conducted by Mallinckrodt Chemical Works, St. Louis, Missouri, that utilize materials obtained under Source Material License No. C-2734 and Special Nuclear Material License No. SNM-33. The inspected activities are confined to one plant located in Hematite, Missouri.
10. The inspection was conducted by Dr. Marvin M. Mann, Division of Inspection, Washington, D. C., and Leo Dubinski and Donald C. Hubbard, Inspection Division, OROO, who, during the course of the visit, interviewed the following licensee personnel:
  - Dr. W. M. Leaders - Technical Director of Special Metals Division
  - Dr. E. D. North - Manager of Hematite Plant
  - Mr. J. W. Miller - Supervisor of Industrial Hygiene Department
11. The licensee currently holds two licenses that cover the Hematite plant operation--one is for source material and the other is for special nuclear material. The licenses have expanded as follows:
  - A. License No. C-2734
    - (1) Issued September 23, 1955, for 500 pounds of UO<sub>3</sub> to be used in UO<sub>2</sub> reactor fuel element studies.
    - (2) Amended November 9, 1955, to allow transfer of UO<sub>2</sub> to other licensees.
    - (3) Amended December 30, 1955, to provide for 500 pounds of UO<sub>3</sub> and 1,000 pounds of UF<sub>6</sub> to be used in the preparation of UO<sub>2</sub> for fuel element studies.
    - (4) Amended March 9, 1956, to provide for 500 pounds of UO<sub>3</sub> and 4,000 pounds of normal UF<sub>6</sub> for fuel element studies.
    - (5) Amended May 24, 1956, to provide for 500 pounds of UO<sub>3</sub> and 5,000 pounds of normal UF<sub>6</sub> for fuel element studies.
    - (6) License expired September 1, 1956.
    - (7) Renewed October 9, 1956, to provide for processing and fuel element studies involving up to 500 pounds of UO<sub>3</sub>, 100 pounds of UO<sub>2</sub>, and 2,000 pounds of UF<sub>6</sub>.
    - (8) Amended March 19, 1957, to provide for 3,100 pounds of normal uranium metal and compounds for fuel element studies, processing, and resale.
    - (9) Amended April 26, 1957, to provide for 500 pounds of UO<sub>3</sub>, 100 pounds of UO<sub>2</sub>, and 7,000 pounds of normal UF<sub>6</sub>.
    - (10) Amended May 24, 1957, to provide for 8,100 pounds of normal uranium metal and compounds to be used for processing, fuel element studies, and resale.
    - (11) Expired November 1, 1957.
    - (12) Renewed October 31, 1957, to provide for 13,000 pounds of normal uranium metal and compounds to be used for processing, fuel element studies, and resale.

- (13) Amended November 22, 1957, to provide for unlimited amounts of normal uranium metal and compounds to be used for processing, fuel element studies, and resale.
- (14) Amended April 14, 1958, to provide for unlimited amounts of normal uranium and thorium metals and compounds for processing, fuel element studies, research and development activities, and resale.

B. License No. SNM-33

- (1) Issued July 18, 1956, to provide for unlimited amounts of uranium enriched up to 7.5% to be converted to UO<sub>2</sub> using procedures set forth in the licensee's letter dated May 14, 1956. The procedures included design information pertaining to plant facilities, shipping containers, criticality control, process equipment; radiological safety procedures; and detailed process procedures.
- (2) Amended August 8, 1956, to provide for unlimited amounts of uranium enriched up to 7.5% and 1.1 kg. of 20% enriched uranium. Use is according to additional procedures dated June 18, 1956.
- (3) Amended August 24, 1956, to provide for unlimited amounts of enriched uranium to be used according to additional procedures dated August 9 and August 21, 1956. The procedures involve essentially the same process as described in earlier correspondence but with some minor equipment and chemical flow-sheet changes.
- (4) Amended February 19, 1957, to allow the preparation of uranium oxide and uranyl sulfate from UF<sub>6</sub> according to procedures dated January 21, 1957. The procedures describe in detail the process, including criticality considerations, for enrichments ranging from 20% to full enrichment.
- (5) Amended April 22, 1957, to include procedures dated March 5, 1957, which consisted of changes in transportation arrangements and design of shipping containers.
- (6) Amended October 28, 1957, to include procedures dated October 21, 1957, that set forth a mode of operation used for the conversion of UF<sub>6</sub> to UO<sub>2</sub> at assays between 3 and 5% enrichment.
- (7) Amended March 3, 1958, to include the preparation of uranium metals and compounds, blending operations and waste recovery as described in three procedure supplements dated February 4, 1958. The procedures cover equipment and techniques used to produce uranium metal of any enrichment and blended batches of UO<sub>2</sub> up to 5% enrichment. Also described were two facilities used for solvent extraction recovery of uranium from scrap material. One facility is designed to handle scrap up to 5% enrichment while the other is designed to process scrap containing from 5% upward to highly enriched uranium.
- (8) Amended April 28, 1958, to include pelleting, additional fuel preparation and waste disposal procedures as described in licensee procedures dated March 14 and April 7, 1958. The March 14 procedure incorporates by reference three applications dated February 20, 1958, and one dated February 25, 1958. The amendment approved a UO<sub>2</sub> pelleting process and facility, an all-assay UO<sub>2</sub> production facility,

processes for producing uranyl nitrate and uranyl fluoride (crystals and solution), and shipping containers for the  $UO_2F_2$  and  $UO_2(NO_3)_2$ .

12. In addition to the source material activities conducted at the Hematite plant by the Special Metals Division, the licensee also carries on two additional source material programs. One program, conducted by the Sales Group under Source Material License No. D-217, is a nonprocessing type activity limited to 900 pounds of source material for resale and use as an analytical reagent. The other program involves the processing of euxenite ore for the Government Services Administration under that agency's Source Material License No. D-195. This process is conducted by the Operations Group in one of the licensee's buildings located in St. Louis. Uranium obtained from the ore is sold to the Commission's Weldon Spring, Missouri, Feed Materials Plant, while the thorium residues are stored at the Army's Granite City Engineer Depot in Granite City, Illinois.
13. One additional special nuclear material license has been issued to the licensee for research purposes to develop a process for producing  $UF_4$  by a direct conversion from  $UF_6$ . All development work for this process will be done at the Hematite plant if the research efforts are pursued.
14. The details of this report cover only the activities conducted under License Nos. C-2734 and SNM-33.

## II. LICENSEE BACKGROUND

15. The Mallinckrodt Chemical Works was one of the first companies to commercially produce uranium and uranium compounds and has been continuously engaged in the field since 1942. The company is a prime contractor of the U. S. Atomic Energy Commission and currently operates the Commission's uranium refinery plant at Weldon Spring, Missouri, and formerly operated a similar plant on Destrehan Street in St. Louis, Missouri.

## III. ORGANIZATION

16. The licensee is a stockholder company organized so as to have seven major operating groups as shown in Exhibit A. The Uranium Division of the company is the group responsible for the contract operation of Commission-owned facilities in Weldon Spring in which refined uranium oxides, uranium tetrafluoride and uranium metal are produced:

Following the 1954 amendment to the Atomic Energy Act, which was designed to encourage private business participation in the atomic energy field, the licensee created the Special Metals Division to further the company's private commercial efforts.

17. The Special Metals Division was formed on January 1, 1956, and placed under the management of Mr. Frederick M. Belmore who holds a B. S. degree in chemistry and a B.S.E. degree in chemical engineering. Mr. Belmore was employed in various capacities by the Manhattan Engineer District and the Atomic Energy Commission from 1943 until 1954. The last Commission position held by Mr. Belmore was that of Deputy Director of the New York Operations Office. In addition to being Manager of the Special Metals Division, Mr. Belmore is also Special Assistant to the President of the company, Mr. Fistere, and the Special Metals Division reports directly to Mr. Fistere. Dr. W. M. Leaders, Technical Director of the division, is also the Technical Assistant to Mr. Belmore, while the Hematite plant operation responsibility is assigned to Dr. R. W. Shearer who reports directly to Mr. Belmore. Dr. Shearer is

assisted by the Plant Manager, Dr. E. D. North, and Dr. G. W. Thompkins who directs research and development efforts at the Hematite plant (see Exhibit B).

18. A. The plant operating force consists of approximately 25 persons working under the supervision of the Plant Manager, Dr. North. Reporting to Dr. North directly are two Process Engineers, who act in staff capacity, and a General Foreman. Other supervision includes the three Assistant Foremen, one to a shift, who report to the General Foreman. In the absence of the Plant Manager and the General Foreman, the Shift Foreman is the senior and responsible supervisor of operations. In addition to the operating personnel there are several other persons such as mechanics, porters, and guards who also report to Dr. North.

The Process Engineers are both graduate chemical engineers who normally work day shift but often work around the clock if a situation arises that requires non-routine hours. Research and development people have on occasions in the past acted as relief for the Process Engineers. The Process Engineers' major responsibilities are to check on personnel to determine if written procedures and standards are being complied with and to review each morning all process reports that were completed on the night shifts. Both men have read Los Alamos and Oak Ridge criticality data reports and have copies available for their use but they are not regarded as criticality experts.

No technical training is required for any of the foremen or operators and generally speaking the foremen are old-time employees who have come up through the chemical operator ranks. Operators start as trainees and progress through B and A operator grades, then utility operator with the next promotion being to that of Assistant Foreman.

- B. The plant has a small analytical laboratory, employing five persons, that is maintained and operated by the operating organization. The research and development laboratory, a separate group, is staffed by several technical persons and technicians from the Research and Development organization of the Special Metals Division who are not responsible to the Plant Manager but report instead to Dr. Shearer through Dr. Thompkins.
- C. It appears that operating personnel have been selected with care and that all supervisors and senior operators have some experience in normal uranium processing. Experience in processing of enriched uranium prior to initiation of the Hematite operation, however, is confined to Dr. Leaders, Technical Assistant to the Manager of the Special Metals Division.

#### IV. FACILITIES

19. The Hematite facility is located on a 150 acre tract of land approximately 40 miles south of St. Louis near the town of Hematite and is bound on the north by a public road, on the south by Joachim Creek, and on the east and west by other private property. The plant is centrally located on the 150 acres and the nearest occupied area is a farm house located several hundred yards away. The production area consists of one main building which, including office space and utility areas, contains approximately 16,000 sq. ft.

On the east side of the main building is the incoming storage and blending building while on the west side is the outgoing storage building. The entire plant area is surrounded by a fence and the gate is manned by guards twenty-four hours a day. A new building duplicating the main building in size is now under construction outside of the fence. When completed, the building will be occupied by both the research and development and operating groups. The fence will be enlarged in the future to include the new building.

20. The main building consists of three separate processing areas that are separated so that materials of various enrichments may be manufactured simultaneously.
  - A. Area number one is designed to handle materials of high enrichment, that is 20% U-235 and higher. The room houses a general products line primarily used for high-fired uranium dioxide for cermet type fuel elements and a process line for the manufacture of metal. Operating in conjunction with the high assay production unit are two auxiliary areas, one containing special equipment used for the solvent extraction of highly enriched uranium from scrap material while the other, a soluble products area, is used for the preparation of crystals or solutions of uranyl nitrate, uranyl sulfate, and uranyl fluoride.
  - B. The second area is for large scale production of low enrichment materials that range up to 5% enrichment in U-235. The main product is ceramic grade uranium dioxide. The equipment in this area, like that in area number one, is also housed in special hoods for dust control and consists of standard chemical plant equipment--tanks, pumps, filter processes, resistance dryers, resistance furnace, etc.
  - C. The third processing area is actually divided into three sections. One section contains equipment for solvent extraction of low enrichment uranium from scrap, while another is for the manufacture of middle enrichment (5 - 20% U-235) uranium compounds. The third section contains the research and development activities and a pilot plant for pressing and firing ceramic pellets of  $UO_2$ . The pelleting line equipment can be used for all assays of uranium and while said to have been initially installed primarily for quality control purposes in manufacturing ceramic grade oxide, it is also used for the production of high density  $UO_2$  pellets for direct loading into fuel elements.
21. The incoming material storage vault is a one-story reinforced concrete building (~30' x 15') equipped with a concrete floor and is located approximately 50 feet to the east of the main building. The room is equipped with chains and brackets that are fastened to the wall and used to secure shipping containers. All shipping containers containing uranium of the same degree of enrichment are chained together. Vault responsibility lies primarily with the process engineers who are assisted by the foremen.
22. The blending building is a single room contained in a concrete block building (20' x 50') located directly south of and adjacent to the incoming storage vault. Blending equipment is contained within a dust control hood and consists of ten 15-gallon open top drums mounted on a rack in back of a roller rail. Each drum is separated from adjacent drums by a one foot slab of concrete.

The south end of the room is used for the storage of containers which are designed so as to give a minimum two foot spacing between adjacent containers.

23. The outgoing storage vault is a small (~20' x 10') reinforced concrete building located approximately 50 feet to the west of the main building. The room is equipped with racks and chains attached to the wall that are used to keep containers separate prior to shipment.

#### V. RADIOLOGICAL SAFETY PROGRAM

24. The over-all radiological safety program of the Hematite plant is under the direct supervision of J. W. Miller, Supervisor of the Industrial Hygiene Department, who reports to Mr. J. G. Moore, Vice-President in charge of Operations. Mr. Miller holds a B.S. degree in chemistry and has had several years' experience in the licensee's Uranium Division's Health Division covering similar normal uranium operations. He is by training both a Health Physicist and Industrial Hygienist and is responsible for all of the licensee's commercial operations, including the Hematite plant. He is assisted by two technicians. Control of factors affecting criticality are not part of the radiological safety program but are handled separately by Dr. Leaders and Dr. North with Dr. Leaders having the prime responsibility.
25. Personnel monitoring is conducted by routine use of film badges, bio-assays, and physical examinations. Film badges are obtained from St. John X-Ray Laboratory in Califon, New Jersey, and are distributed to personnel on a frequency determined by individual job assignment. Permanent operating personnel at the Hematite plant are monitored on a weekly basis while rotating personnel are monitored monthly. Complete individual exposure records and summaries are maintained on all employees and were reviewed. The average exposure of Hematite plant personnel during the past year, as indicated by the records, was 80 mrad due to beta radiation and 36 mrad due to gamma radiation. The maximum single six months accumulative exposure recorded as due to beta was 2525 mrad while the maximum due to gamma was 380 mrad. No single weekly exposure in excess of the permissible weekly exposure was noted. Film badges are worn by all persons entering the Hematite plant and are distributed and collected by the guard at the door.

Clinical and radiological urinalyses are made routinely with the frequencies determined by individual job assignment. The minimum is one analysis per year while the maximum number of routine analysis per person is four per year. The radiological urinalyses are performed by a commercial organization, Nuclear Service and Engineering Corporation, located in Pittsburgh, Pennsylvania. This service was formerly supplied by the Tracerlab Corporation. Results are reported in disintegrations per minute per liter (~24 hour sample) with 46 d/m/l established as tolerance. The maximum single exposure, as indicated by the licensee's records, was 37.4 d/m/l while the average during the past year was of the order of 5 d/m/l. Pre-employment physicals are given all employees.

Protective clothing, including shoes, are provided and laundered by the company in their own laundry facility. A divided change room is used by employees to prevent street clothing from being carried into areas where there is a possibility of contamination.

26. The licensee's airborne radioactivity control program operates on the philosophy that a correctly designed installation will control the material in such a manner that the material does not become a health hazard to the employee who operates the installation. Thus, maximum attention is given to adequate engineering design and to the development of standard operating procedures which will provide acceptable control. Dust studies (general and breathing zone) of individual jobs are made to determine an operator's exposure to airborne material while carrying out his normal duties. Based upon the dust studies, changes are made to achieve the lowest practical exposure. Concentrations greater than the plant's maximum permissible concentration ( $70 \text{ } \mu\text{d}/\text{m}^3$ ) during some operations may be permitted provided the weighted average for the daily job does not exceed one M.P.C. In general, design and operation are aimed toward an upper limit of 1/10 M.P.C. Once a job has been evaluated, it is assumed that the exposure of each operator on that job will be approximately the same. The exposure value for that operation is then charged to each person who performs the operation. A complete job history is maintained on each employee and a re-study of each operation is made on a periodic basis to establish an up-to-date exposure value. High urinalysis results and film badge reports also require a re-evaluation of an operation.

Air samples have been taken on the roof of the main building with the maximum results noted being  $1 \times 10^{-12} \text{ } \mu\text{c}/\text{ml}$  of air. Samples have not in the past been taken outside of the fence or at the outer edge of the property line, but plans are being developed to do so in the near future.

All dusty operations are enclosed in hoods equipped with double filters, and, in addition, all personnel are provided with face masks which are worn around the neck when working with a dust generating process even though the process is enclosed in a hood.

Complete air surveys are made of the entire plant at least twice a year and some sampling is done in the plant at least one week out of every month; however, no full-time Health Physicist is assigned to the Hematite plant.

27. Area monitoring of the entire plant, using survey meters, is done at least four times a year formally and more often informally. The smear technique for detecting contamination is not used. The results of the four formal surveys are recorded on floor plans which are filed in Mr. Miller's office. The records available were reviewed with no significant radiation levels noted; however, it was pointed out to Mr. Miller that both his area monitoring and air sampling survey records were considered to be close to marginal since they did not present a complete picture of the Hematite operation. He stated that the program would be expanded in the future in order to fill in the gaps noted in his present records. It was also pointed out that instrument surveys are not always adequate for revealing low level alpha contamination and that since he had counting equipment available he might want to evaluate the use of smears for that purpose.
28. Instrumentation for the radiological safety program consists of:
- 2 - Technical Associates Model No. 3 Juno ( $\alpha$ ,  $\beta$ ,  $\gamma$ )
  - 3 - Victoreen "Thyac" Model 389C Survey Meter ( $\beta$ ,  $\gamma$ )
  - 3 - Victoreen Model 356 Survey Meter ( $\alpha$ ,  $\beta$ ,  $\gamma$ )
  - 2 - Nuclear Measurements Corp. Model PC-3A Proportional Counters ( $\alpha$ ,  $\beta$ ,  $\gamma$ )
  - 8 - Portable Gast Air Samplers (1/3 H.P. motor)

29. Most of the contaminated liquid waste generated by the licensee's activities consists of ammonium diuranate. These solutions are currently treated with lime to precipitate the uranium and fluoride values then the slurry is brought to the boiling point to release the ammonia while the precipitate is filtered to remove the calcium fluoride and uranium contents. The filter cake is stored for future processing while the filtrate which is essentially pure water is released to the process waste sewer line. The ammonium fluoride solution analyses have shown the maximum uranium contents to be 50 parts per million with the average value between 5 and 25 parts per million. Ninety-nine per cent of the ammonium fluoride liquors are produced in the section of the plant that handles up to a maximum of 5% enriched uranium. Since the uranium contents are so low the licensee in the near future proposes to stop the lime treatment and transport the untreated ammonium fluoride by truck to its main plant in St. Louis where the material will be used in another process. This procedure has been approved by the Division of Licensing and Regulation.

Liquid effluent monitoring of the Hematite plant discharge is conducted at several locations on a non-routine basis using a grab sample technique. All process wastes are discharged due west from the main building through a sewer that empties into a stream running south through the licensee's property. From there it flows several hundred feet then discharges into the Joachim Creek which is not on licensee's property. Samples have been obtained from the process sewer, from process filtrate, in the stream, and at several points in Joachim Creek, both below and above the entry point of the licensee's stream. Effluent sampling records were reviewed and the highest concentration noted was  $6.03 \times 10^{-6}$   $\mu\text{c/ml}$  of alpha activity which was detected in the filtrate. The highest concentration reported off the licensee's property was detected in Joachim Creek just below the licensee's point of entry. This was detected on July 27, 1957, and showed a concentration of  $1.84 \times 10^{-7}$   $\mu\text{c/ml}$  of alpha activity.

It was pointed out to Mr. Miller and Dr. Leaders that with the company's increase in production during the past year it would appear that additional and more recent effluent information obtained on a more routine basis would be desirable for their records. Mr. Miller stated that there were some additional sample results recorded but that he was unable to locate them during the visit. He also stated that the stream on the company's property will be monitored with a continuous sampler in the near future. No figures were available as to the average volume of the plant's liquid effluent or the average flow of Joachim Creek.

30. Shipping containers are monitored by a Health Physicist to assure compliance with ICC regulations and all containers are posted externally with ICC type labels.
31. Regulation type posting is in use throughout the plant including each individual container of material. A color coding system has been combined with container posting which identifies the enrichment of the material in the containers.
32. As a result of the Oak Ridge (Y-12) criticality incident, the licensee is now in the process of obtaining a radiation detection and alarm system that will be installed at several points throughout the plant. A system designed by the Radiation Counter Laboratories is currently being considered.

33. Radiological safety education and instruction of personnel has been conducted on a limited scale since most of the operating personnel now employed have worked either in the company's Uranium Division or the Euxenite Plant. Several lectures are said to have been given by Dr. North on a non-routine basis that were concerned with housekeeping items observed by him during tours through the plant. Written instructions consist of one four-page memorandum entitled "Health Procedures - Hematite Plant" written by Mr. Miller and distributed to top supervision. Copies were observed posted on bulletin boards located at several prominent points within the main building.

#### VI. PROCESSES

34. The main process of the plant is one whereby uranium dioxide ( $UO_2$ ) is produced from normal and enriched grade hexafluoride ( $UF_6$ ) through an intermediate diuranate (ADU) step. Although the scale of equipment is different depending on whether high or low enriched material is being processed, the basic flow sheets for the production of ADU type  $UO_2$  are identical. Basically, the process consists of the hydrolysis of  $UF_6$  with a dilute ammonia solution to give a precipitate of ammonium diuranate which is filtered, washed, and dried. The dried powder is pyrohydrolyzed with steam and at the same time the ADU is converted to black oxide ( $U_3O_8$ ), which provides an intermediate that is used for preparing other uranium compounds. The  $U_3O_8$  is then reduced to  $UO_2$  with hydrogen or cracked ammonia. If the  $UO_2$  is to be pressed into pellets for use in fuel elements it is blended, pelleted, and sintered at high temperatures.  $UO_2$  high fired crystals and ceramic grade  $UO_2$  may also be produced following the blending step or the  $UO_2$  may be converted to green salt ( $UF_4$ ) for use in preparing uranium metal. Although Mallinckrodt is licensed for processing thorium, none has been obtained to date.

Process scrap, including such items as filter bags, clean-up scrap, rejected pellets, destructive test samples, and analytical scrap, is dissolved in acid; following this the uranium values are extracted in solvent then put in a purified uranyl nitrate solution which can be converted to ADU. Scrap from the processes is recovered from the hoods, filters, and equipment and returned to the customer.

#### VII. MATERIAL ACCOUNTABILITY

35. Adequate material accountability procedures are in effect. Source and special nuclear materials are received in cylinders as  $UF_6$  which are shipped to the Hematite plant by private commercial truck lines. When the trucks arrive, the cylinders are unloaded, weighed, and stored under the supervision of the foreman who enters the number and contents of each container in a log book kept in Mr. North's office. Cylinders are removed only when they are to be put into the process and are transferred according to number at the direction of the foreman or assistant foreman. Once material from a cylinder is put into the process it is continuously being weighed and accounted for and the amounts remaining after each step are recorded on detailed process flow sheets which are reviewed each day. The flow sheets used along with the regular accountability records serve as a double entry system of accountability. The ultimate accountability responsibility rests with the Technical Director, Dr. Leaders, who prepares and submits periodic reports to the AEC on Form #578. Dr. Leaders is also responsible for all

shipments from the Hematite plant and he carefully checks all users to make certain that they are either valid licensees or else are exempt from licensing.

36. The amount of source and enriched grade uranium processed and shipped by the licensee each year is quite large. For example, during 1957 the following amounts of material were handled:

A. Source Material

UF <sub>6</sub> received	11,901.98 lbs.
UF <sub>6</sub> used	11,038.75 lbs.
UF <sub>6</sub> on hand (1-1-58)	863.23 lbs.
UO <sub>2</sub> purchased	8,306.76 lbs.
UO <sub>2</sub> shipped	1,247.88 lbs.
UO <sub>2</sub> on hand (1-1-58)	
Research Department	349.67 lbs.
Warehouse	6,634.90 lbs.
Residues (U content unknown)	156.92 lbs.

The licensee has found it uneconomical to completely empty the source grade UF<sub>6</sub> cylinders, and, as a result, each cylinder returned to the Commission's Paducah, Kentucky, plant contains approximately 1.5 pounds of UF<sub>6</sub>. In arriving at an over-all material balance in the above figures the licensee charged themselves with the total weight of UF<sub>6</sub> recorded in the original transfer papers though it is estimated that the returned UF<sub>6</sub> is approximately 1/4 of 1% of the amount received.

B. Special Nuclear Material

(1) Received

UF <sub>6</sub> (2.3%)	1877.66 lbs.
UF <sub>6</sub> (2.7%)	6936.68 lbs.
UF <sub>6</sub> (1.5%)	4721.604 lbs.
UF <sub>6</sub> (5.4%)	16.24 lbs.
UF <sub>6</sub> (6.8%)	11.257 lbs.
UF <sub>6</sub> (20%)	308.216 lbs.
UF <sub>6</sub> (90%)	149.950 lbs.
UF <sub>6</sub> (NS-40)	362.359 lbs.

(2) Shipped

UO <sub>2</sub> (2.3%)	1534.0 lbs.
UO <sub>2</sub> (2.7%)	6722.63 lbs.
UO <sub>2</sub> (1.5%)	2589.68 lbs.
UO <sub>2</sub> (5.4%)	16.24 lbs.
UO <sub>2</sub> (6.8%)	11.034 lbs.
UO <sub>2</sub> (20%)	173.784 lbs.
UO <sub>2</sub> SO <sub>4</sub> (20%)	57.571 lbs.
U <sub>3</sub> O <sub>8</sub> (20%)	68.601 lbs.
UO <sub>2</sub> (90%)	134.172 lbs.
UO <sub>2</sub> SO <sub>4</sub> (90%)	4.704 lbs.
UO <sub>2</sub> (NS-40)	345.54 lbs.
UF <sub>6</sub> (2.3%)	283.32 lbs.

the procedures and standards were undergoing review and would be revised to conform with experience gained during the past months.

38. Once a process is turned over to the Production Group, the foremen and process engineers assume primary responsibility to Dr. North for monitoring the process to ensure that the operations are carried out according to the developed procedures. These men are aware of the criticality limitations for each step of the process and for each piece of equipment and are responsible for seeing that the operators do not exceed them. The foremen and engineers are continuously circulating in the areas during the production and handling of source and enriched grades of uranium. Operator process flow sheets are reviewed at least once daily to ascertain that procedures are being followed. All operators are said to have been instructed to contact the foremen or engineers if they are uncertain about any phase of their work or if there appears to be an unusual situation arising.
39. Material is received and shipped in approved "always safe" containers that utilize the volume limitation and physical separation principles to prevent the accidental accumulation of a critical mass. These "bird-cage" containers are also used for storing the raw material as well as the finished product. Movement of material within the building is normally done by hand and is usually carried out within dustproof hoods that are physically divided. Foremen are said to control and direct all movements of material in order to prevent more than one container from being in motion in one area at the same time. Equipment in each of the process lines has been so dimensioned as to be "always safe" for the intended enrichment of material. It appears that thorough and conscientious use of experienced consultants has been made in an effort to ensure adequacy of design. All vessels, retorts, trays, and pipes are so sized as to enjoy a factor of safety of approximately two or greater, the factor being based on Oak Ridge and Los Alamos data.
40. It appears that the plant equipment has been carefully designed to minimize the probability of accidental accumulation of critical masses of fissionable materials. There are, however, a number of points in the processes and in the laboratory activities wherein mistakes could occur and critical masses could be assembled. The ones noted are as follows:
  - A. The raw material for the several processes is  $UF_6$ . This material is received from Oak Ridge in "always safe" containers for the material involved. The selection of appropriate containers from storage and the introduction of material to the process line usually depends upon one person. Records of containers and their contents are kept in the Manager's office and the Manager's written approval is required before removal of a container from storage, but the actual removal and placement of material into the process depends upon correct action by this one person. It does not appear that a procedural or analytical check exists for this operation; therefore, the introduction of highly enriched material into the processes for intermediate enrichments is possible.
  - B. At several points in the processes, "always safe" trays are carried by hand from one piece of equipment to another, and, in some cases, trays of enriched material are stored temporarily on tables. Safety in these operations is clearly dependent on proper actions by individuals.

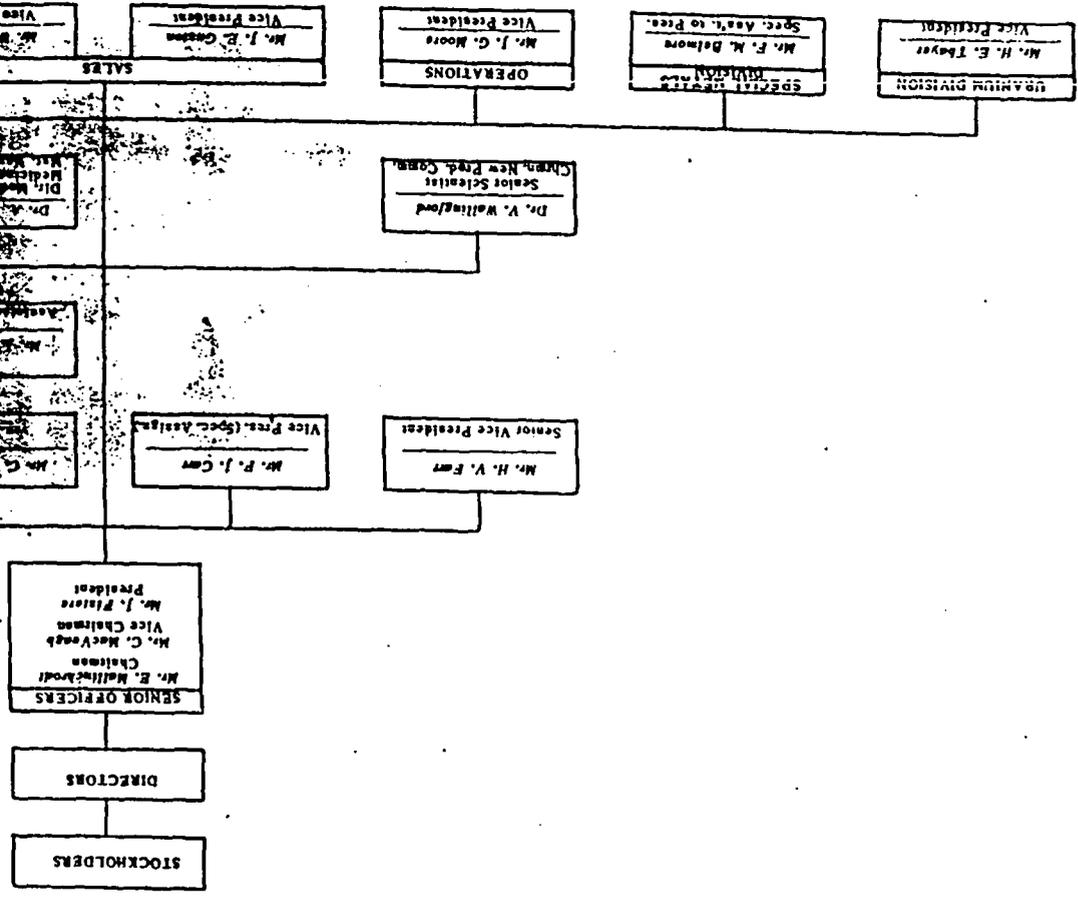
- C. It was observed that in connection with research and development activities, bottles containing uranium solutions are stored temporarily on shelves without physical limitation on geometry and quantity. At the time of inspection, each of some three bottles grouped on a shelf were observed to contain more than 100 grams of Uranium 235 in solutions of greater than 20% enrichment.

After the operations had been reviewed, a conference was held with Dr. North, Plant Manager; Dr. Leaders, Technical Director; and Mr. Miller, Industrial Hygiene Supervisor. The specific items noted above were discussed in some detail and the inspection representatives pointed out that by and large the operation is considered as being well designed and responsibly supervised. However, it was noted that, in the opinion of the Division of Inspection, reliance upon individuals at certain points in the process presents a potential hazard and that improvement in operational safety could possibly be realized by further development of procedure and procedural checks. The subject of divided responsibility in regard to research and development activities was mentioned, particularly since it had been observed as noted in section C above that certain questionable procedures were being followed by research personnel. It is the understanding of the Division of Inspection that Mallinckrodt management is taking steps to improve the situation.

#### IX. SUMMARY

41. The use of source and special nuclear materials in the activities conducted by Mallinckrodt Chemical Works at the company's Hematite, Missouri, plant are in accordance with the license conditions, license applications, and license application supplements.
42. Plant supervision appears to be composed of experienced and qualified personnel who are interested in conducting safe nuclear and radiological programs in conjunction with the licensee's activities. The radiological safety program under Mr. Miller's direction is functioning effectively but additional survey and effluent monitoring information appears to be desirable. A more complete program covering these points is said to be planned for the future. The nuclear safety program under the direction of Dr. Leaders, while adequate from the equipment design point of view, does involve activities at several points in which there is reliance upon individuals to carry out procedures. Any reduction in the procedures which would limit reliance on individuals would further improve the over-all safety of the operation. Management intends to continuously review these matters and make improvements where reasonably possible.
43. It appears that Mallinckrodt management, immediately related to the Hematite operation, understands fully the questions raised by the Division of Inspection and intends that the operation shall be conducted safely.
44. No specific recommendations are made at this time.

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MALLINCKRODT CHEMICAL WORKS	NEW	Organization Chart	



Organization Chart  
Special Metals Division  
of  
Mallinckrodt Chemical Works  
August 1958

