#### PART 40 INSPECTION

A-29

Rare Earths, Inc. Division of W. R. Grace & Co. Pompton Plains, New Jersey

Date of Inspection: November 25, 1959

#### Persons Accompanying Inspectors

Mr. John Russo, New Jersey State Department of Health

#### Persons Contacted:

Richard Mandle, Plant Manager Richard Stone, Sales Manager D. Hubbard, Manager, Industrial Relations, Erwin Plant, Davison Chemical Company

#### DETAILS

### License #R-196 (Items 9 thru 20)

#### 9. Introduction

On November 19, 1959, John Russo, New Jersey State Department of Health, telephoned this office to inform us that on June 11, 1959, several members of his department were taking routine water samples in the Wayne-Pompton Plains area in New Jersey, when they noticed a milky dispersion in the Pequonnoch River. Samples taken and analyzed of this dispersion revealed alpha contamination of 3370 uuc/l and beta contamination of 1495 uuc/l. Russo stated that approximately 1/4 mile upstream from the sampling point is located Rare Earths, Inc., Division of W. R. Grace & Co. He stated that he and his associates toured the plant and found that the plant was processing monasite mand. He noted that there was approximately 9000 lbs. of ThO2 stored in their backyard and when it rained this material was being washed down the river. He added that Rare Earths, Inc. had been taken to court last year, convicted and fined for general pollution of the area and the river surrounding the plant.

#### 10. Organization and Procedures

Rare Earths, Inc., a branch plant of Davison Chemical Co., a division of W. R. Grace & Co., is engaged in the manufacture of rare earths oxide (Re203) from monasite sands containing from 3 to 3.5% thorium oxide.

Richard Mandle is the plant manager, while Richard Stone is the sales manager. Mandle reported that he is the radiological safety officer (RSO). Mandle stated he has had no formal training in radiation protection. He said he attended several lectures at Brookhaven in 1949 relative to rare earth processing and obtained information on radiation protection and monitoring. He noted that he attended a lecture on radiochemistry given by John Harley, HASL, NYCO.

D. Hubbard, Manager, Industrial Relations, Erwin Plant of Davison Chemical Co., a division of W. R. Grace Co., stated that he had some up to the Rare Earths plant for the first time on November 24, 1959 at the request of Mandle so that he (Hubbard) could be present during the inspection. Hubbard has a BA degree in physics and law degree from Vanderbilt. He was employed as a health physicist for the Union Carbide & Carbon Co., in Oak Ridge, for approximately 12 years and for the AEC as a member of the OROO Inspection Division for approximative years.

Mandle stated the plant was on a 24-hour day operation, and that the twenty-five employees were composed of seven production workers and ten office employees on and 8 to 4 shift, two production workers on a 4 to 12 shift, two workers on a 12 to 8 shift, and three maintenance men. So minors are employed.

#### 11. Facilities and Uses

A two story brick building containing a production area (approximately 8000 square feet), three quality control labs (1200 square feet), and offices are located in Pompton Plains, New Jersey. The plant facilities are located on Black Oak Ridge Road (Rt. 202), a main thoroughfare. The production area consists of monasite ore storage, ball will, filter press, rare oxide, cloride, and thorium refining areas. Alapout of the plant is included in the licensee's file. Facilities for change lockers, kundry, and lunch room are available for the production personnel. Mandle wished to have the layout and process description treated as "business confidential". A waste treatment facility and several waste storage tanks, Th(OH) drum storage and several areas where process and waste sludges were stored in open piles are located outside the plant. Mandle supplied a brief description of the operations involved in the processing of Re2Og from monasite sand. The process description which includes the location and type equipment used follows:

#### \*FIRST OPERATION - Digestion of the Monagite (Sulfonation Reactor)

The first operation of the process involves digestion of the finaly ground monasite sands with hot concentrated sulphuric acid. The rate of the reaction of monasite sand with sulphuric acid, or sulfonation, increases with finer particle size of the monasite sand and higher reaction temperatures. The reaction starts as a fluid mixture of the two components. As the reaction proceeds it gradually becomes more viscous and finally putty-like due to the formation of voluminous applydrous rare earth sulfate crystals. The phosphate content of the monasite goes into solution as phosphoric acid. Further agitation will cause sufficient thinning of the mixture, to allow discharge from the cast iron reactor. The reaction may be considered complete at the end of 4 to 6 hours.

#### SECOND OPERATION - Crystallisation (Tank 1, Centrifuge & Press 5)

The second operation involves the crude separation of the thorium sulfate from the rare earth sulfate. At the end of the sulfonation reaction, the hot charge is quenched in a tank containing recycled acid and wash streams from subsequent process steps. The wash streams contain sufficient water to dilute the free acid in the sulfonation to approximately 50% total acidity, and also provide water hydration for rare earth sulfates from sulfonation.

The hydrated rare earth sulfates form as a dense crystalline salt in a slurry of approximately 50% phosphoric sulphuric acid liquor. The thorium sulfate produced in the sulfonation is more soluble in this acid than the rare earth sulfates which permits a crude separation of thorium and rare earths.

The hydrated rare earth sulfates from the crystallization are pumped to a classifier to remove the finely ground non-monasite gangue and acids from the rare earth sulfates. The overflow from the classifier is filtered through a precoat drum filter to separate the gangue from the thorium-rich acid liquors. A portion of this filtered acid is removed for thorium separation and the remainder is recycled to the crystalliser tanks.

THIRD OPERATION - Rare Earth Removal from Acid Stream (Tank 24, Press 54, Tank 15)

The thorius—rich acid liquors, or top acid, contain a small quantity of the original rare earths contained in the monasite. These rare earths are stripped from the acid by the addition of sodium sulfate which forms an insoluble acid rare earth double salt. This double salt contains some occluded thorium and therefore must be processed to properly distribute the rare earth and thorium values. The double salt is separated from its acid liquor, called stripped acid, by means of a drum filter. The acid rare earth double salt is converted to water insoluble rare earth hydroxide by treating it with boiling caustic sods. The caustic soda and soluble salts are removed by hot water washes and the thickened rare earth hydroxide is then mixed with the washed rare earth sulfate crystals in operation 6.

FOURTH OPERATION -Thorium Separation from Acid Stream (Tank 25, Press 5B, Tank 16, Filter 3)

The thorium is removed from the stripped acid by addition of either sodium fluoride or hydrofluoric acid which causes insoluble thorium fluoride to precipitate from the acid. The thorium fluoride is separated from the acid on a drum filter and the spent acid is sent to an acid dilution boot for the Superphosphate Plant. The thorium fluoride is then treated with caustic soda to convert the thorium fluoride to hydroxide. Sodium fluoride and free caustic and removed by water washing in the Shriver thickener. The washed product is then dried and packed as thorium hydroxide product.

FIFTH OPERATION - Removal of Acid from Crude Rare Earth Crystals (Centrifuge, Tank 19)

The hydrated rare earth sulfate crystals from the underflow of the classifier (operation 2) are filtered on a pan filter and counter-currently washed with the rare earth process wash liquors before these liquors are sent to the crystallizing tank. This operation serves to remove the bulk of the phosphoric acid and sulphuric acid from the rare earth crystals so that they may be dissolved in water in operation 6 with a minimum acid contamination since acid interferes with the thorium separation.

SIXTH OPERATION - Removal of Thorium from Rare Earths (Tank 19, Press 1, Tank 6)

The thickened rare earth hydroxide from operation 3 is mixed with the washed rare earth crystals from operation 5 and filtrate from operation 8. The rare earth values go into solution as neutral rare earth sulfates, and gangue and therium remain insoluble as throium phosphate. Complete removal of therium from the rare earths is accomplished by maintaining the pH of this solution at 5.5. The phosphate cake is removed by filtration and the polished rare earth liquors are sent to the second crystallizing tank (operation 8).

SEVENTH OPERATION - Recovery of Thorium and Rare Earths from Gangue (Press 1, Tank 8, Press 6, Tank 21)

The thorium phosphate cake in operation 6 is combined with the gangue from the precoat drum filter in operation 2 and is countercurrently treated with a dilute sulphuric acid solution to solubilize the rare earth and thorium values leaving insoluble residues. These residues are of two types; one consisting of heavy minerals and unreacted monasite; the other consisting of inely divided silica, calcium sulfate, filter-aid, etc. The heavy minerals and monasite are recovered as the underflow of a cyclone classifier and the finely divided material is removed by filtration, and after washing is sent to the dump. The acidified rare earth and thorium liquors are recycled as washes through the crude rare earth crystal filter to the sulfonator crystallization tanks.

# EIGHTH OPERATION - Formation of Rare Earth Double Sulfate (Tank 6, Tank 3, Press 2, Tank 10, Press 7)

In the double sulfate precipitation tank, neutral rare earth sulfate liquors from operation 6 are treated with sodium sulfate to form rare earth double sulfates. This salt forms as a dense precipitate and is removed from the slurry by settling and filtration. The filtrate is collected and treated with soda ash to pHS. which causes the soluble yttrium earths to precipitate. The yttrium earths are filter pressed and stored, the filtrate from the operation goes to the plant waste.

The double salt may be treated with the following for the preparation of rare earth products:

- a) Hydrofluoric acid to give rare earth fluoride.
- b) Caustic soda to form rare earth hydroxide.
- c) Soda ash to form polishing powders.

Rare earth chloride, cerium products and didymium earths are produced from rare earth hydroxide. Heavy rare earths are recovered from yttrium residues."

#### 12. Procurement Procedures and Control

R. Mandle is responsible for ordering monasite sand containing 3 to 3.5% thorium from producers and distributors licensed by the AEC. Mandle reported, to date, Lindsay Chemical Co., West Chicago, Illinois, and Baumhoff-Marshall, Inc., Boise, Idaho, have been his suppliers. Records of purchase orders from both companies are included in the licensee's file.

#### 13. Instrumentation

At the time of inspection an inoperable Beckmann MI-5 beta-gamma survey meter was found to be on hand. Mandle stated that on several occasions a Victoreen alpha survey meter had been borrowed from Ledoux Co. Subsequent to the inspection, R. Stone contacted this office (December 28, 1959) and stated that the instrument has been repaired and that his company intends to procure additional instrumentation.

#### 14. Radiological Procedures and Control

## A. Instructions and Personnel Protection

According to Mandle, production workers have been orally briefed on radiological health safety by F. Nonamaker and himself. A copy of the lecture given to production workers by F. Nonamaker dated April 17, 1958 is included in the licensee's file. The lecture was attended by all workers who were required to sign the sheet of attendance. All production workers, according to Mandle, are equipped with orlon uniforms, respirators, gloves, and rubber overshoes.

#### B. Surveys

No sir surveys for both thoron and thorium have been made to date by the licensee either in-plant or out-plant. No stack air surveys have been made to date. At the time of the inspection, little or no production operations involving the handling of monarite cands or packaging of

of ThO2 and ThOH were in process. Three emples taken at the ReO2 waste press area, mesotherium area, and monasite storage area shised therium concentrations of 2, 60, and 20 alpha d/m/M, respectively. Smear samples taken at various locations inside the plantemed levels of 120 to 540 alpha d/m/100 cm<sup>2</sup>. A 1-1/8" Whatman filter paper pressed on waste silica press cake in the mesotherium area showed a concentration of 190 alpha d/m/1-1/8" filter paper sample. A copy of the air and smear results analyzed by HASL is included as Exhibit A in the report details.

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Mandle reported that two direct radiation surveys have been made to date. Records of surveys conducted on January 6 and Marsh 7, 1959 are included as Exhibits B and C, respectively. Neither survey record includes the instrument used nor the distances from the sources of radiation. Only the January 6, 1959 survey expressed the results in mr/hr. The surveys did not include the radiation levels in the unrestricted outside storage dumps, where piles of yttrium sludge, silica waste (mesothorium containing material), waste treatment sludge and phosphate sludge were stored. The surveys did not include evaluation of Th(OH) drum storage area, where a measurement of 7.5 mr/hr was found at one foot from the drums using a GM survey meter. Other measurements taken by the inspector using a Juno alpha-beta-gamma survey meter #5666, and a Nuclear Measurements Corp. beta-gamma survey meter #5571, which were calibratedoon November 4, 1959 are as follows.

#### LOCATION a. Inside Plant

#### JUNO

#### GM

1. Waste silica press - 80 contact with floor cm

800 alpha d/m/100 cm<sup>2</sup> - 8 mr/hr gamma

2. Passageway to office next to press 50,000 alpha d/m/100 cm<sup>2</sup> - 5 mr/hr gamma 15 mr/hr beta

 Hand wheel between tank #2 and tank #6 6000 alpha d/m/100 cm - 12 mr/hr gamma

4. Storage area

7 mr/hr at 1° from monasite storage bags -1.5 mr/hr bkgd of storage area (waist high meas.)

5. Th(OH) drum storage near tank #12

7.5 mr/hr at 1° from drums

Dut Plant	<b></b>			
1. Gray Pile (Phosphate cake)	ll mr/hr at 1' from pile			
2. Silica and Mesothorium	15 mr/hr at 1' from pile			
3. Yttrium Pile	1.5 mr/hr at 1° from pile			
4. Waste Treatment Sludge	1-2 mr/hr at 1° from sludge			
5. Background bet. Th(OH) drum storage and Yttrium sludge pile	1.3 mr/hr			
6. Th(OH) drum storage area (approximately 30 drums)	12 mr/hr at 1° from drums			
7. Primary mixing tank out- side waste treatment plant	1.5 mr/hr at contact with tank			
8. Waste treatment plant (bkgd)	0.3 - 0.5 mr/hr			

CM

## C. Medical

Mandle stated the preoperational physicals and yearly physicals which include chest I-rays, blood, and check of physical well-being are provided for all plant employees. No urine analysis program is in effect.

## D. Personnel Monitoring

Weekly film badges supplied by St. John I-Ray Laboratory, Califon, New Jersey, are provided employees. The badges also contain a film for a 13-week cumulative exposure. The weekly film badge results for 1958 and 1959 average 100 mr gamma or less while the beta exposures range up to 285 mrep. The 13-week cumulative exposures averaged approximately 1200 mrem. No dosimeters or ring badges are employed.

## 15. Storage and Security of Material

Initially, Mandle declared his entire plant and surrounding ground as his restricted area. However, after a tour of the plant and grounds, he noted that a 4° wooden fence surrounding his grounds did not completely enclose his plant grounds. He then said that his restricted area would be limited to his production plant and waste disposal which was located approximately 75° from the plant. The waste disposal plant was under lock and key. It was pointed out to Mandle that even if the 4º fence covered the entire rear portion of their plant grounds, no control or gated area was available to keep the public from entering the plant grounds, from the parking area, or from the unferced opposite side of the plant, which is located on a main thoroughfare. Handle agreed this was a correct statement. Stored on the unrestricted plant grounds were piles of thorium bearing sludges, i.e., yttrium sludge, silica sludge (mesothorium), phosphate sludge, and waste treatment sludge. Also stored outside the plant in the unrestricted area were over 30 drums of Th(OH). These drums contained an average of 500 lbs. of material. A radiation measurement made with a Juno showed 12 mr/hr at 1' from the Th(OH) drums.

#### 16. Inventory

On hard as of November 15, 1959 was 16,645.4 lbs. of monasite or approximately 5000 lbs. of ThO2. ThO2 residues in barrels totalled 5180 lbs. Thorium content of sludges stored on plant property are as follows:

- a) Ore tailings [gangus (mesothorium)] 230 tons residue containing 8,200 lbs. of ThO2 located in Area G.
- b) Yttrium sludges 200 tons residue containing 3000 lbs. of ThO<sub>2</sub> - located in Area H.
- c) Reworked sludges 137 tons residue containing 2750 lbs. of ThO<sub>2</sub> - located in Area I.
- d) Waste treatment sake 105 tons residue containing 1300 lbs. of ThO2 - located in Area J.
- e) In process silica sludge 30 tons residue containing 2700 lbs. of ThO<sub>2</sub> located in Area H.
- f) In process thorium carbonate 31 tons residue containing 3100 lbs. of ThO2 located in Area L.
- g) In process thorium Hydraxide 15 tons residue containing 10,500 lbs. of ThO<sub>2</sub> located in Area K.
- h) Refined yttrium concentrate 20 tons residue containing 2700 lbs. of ThO2 located in Area M.

A copy of the facility layout which includes the locations of the production and waste treatment plants, sludge storage, and drainage trench constructed by the licensee is included as Exhibit D.

### 17. Waste Disposal

The waste treatment plant treats all liquid wastes issuing from the plant. The waste involved consists of wash water, floor washings and surface run-off from the adjacent plant property.

The process involves the use of an average of 35,000 gallons of water per day. All of the washes are discharged into a common 1000 gallon sump, equipped with two automatically controlled force pumps, which pump the waste to a retention tank. Each pump has capacity to handle the peak load, and is installed so that the second pump starts in case of extreme demand or failure of the first. Signals are installed in a control house to indicate the proper functioning of the pumps.

The retention tank has a capacity of 50,000 gallons, which provides 24 hours average retention of the wastes. In addition to the purpose of acting as a reservoir, or constant head installation, the tank provides means of diluting effluents of widely varying pH so that the automatic pH controlling equipment may function more efficiently. The incoming wastes flow through a distributing channel in the tank, and effluent, after initial settling, is removed from the midpoint of the tank and flows by gravity to a mixing tank. A draw-off is provided at the bottom of the tank to pump accumulated solids to the sludge filter press.

An 8000 gallon mixing tank, equipped with a gate agitator, receives effluent from the retention tank at is midpoint. A pH electrode assembly is in circuit with the mixing tank, and is electrically commected to a mechanically operated diaphragm valve. Two storage tanks are provided to feed either 50% sulphiric acid or 50% caustic sods solution through the automatic diaphragm valve to the mixing tank, as called for by the pH controller. Again, signals are provided to indicate proper functioning of the valve and chemical supply tanks as well as a recording chart which indicates the pH of the mixing tank. The mixing tank effluent is piped to a 2000 gallon Hardinge thickener at pH 5.8 - 6.2.

Mandle stated that no liquid effluent samples were checked by his company to determine the thorium concentration of the liquid effluent discharged to the storm sewer. He said the State had made some checks and that he was going to make arrangements with the State to analyze some water samples for him. No approval to dump liquid effluents into a storm sewer by the AEC was reported to be given his company.

The Hardinge thickener provides a clear overflow to a final clarification tank and adjusted to give a 20% solids underflow which is pumped to a sludge filter press in the control house.

The final clarification tank of 50,000 gallon capacity, provides an average 24 hours of retention time for the effluent before discharge from the system. The main function of this tank is to provide sufficient time for post precipitation of solids after pH adjustment. A draw-off is provided at the bottom of the tank to pump accumulated solids to the sludge filter press.

The sludge filter is of the plate and frame type, with a capacity of 6 cubic ft. of cake. Approximately 60 cubic feet of sludges, or 3500 lbs., are removed weekly. These sludges are hauled to a dump on the property.

The system was designed to operate automatically. Twelve man hours per day are devoted to the maintenance, cleaning and control of the operation. The entire operation is under the supervision of the plant chemist who checks the performance of the equipment, and samples prepared by the shift operator.

Mandle stated, and it was noted during the inspection, that a drainage ditch was under construction to collect run-off water from the hill surrounding the upper end of the licensee's grounds. Another drainage ditch is being constructed between the piles of sludge and the production and waste treatment plant. This, according to Mandle, would prevent run-off to the street.

Mandle said that on several occasions due to the fluxuation in pH, there were slug discharges to the storm sewer. This discharge consisted of both soluble and insoluble wastes (milky white dispersion noted in item 9 of report details).

A telephone conversation with John Russo, New Jersey State Dept. of Health, on January 18, 1960, revealed the following information with regard to release of soluble and insoluble effluent to the streams by the licensee. Russo said on January 17, 1967, a sample taken from the creek near the plant showed a thorium concentration of 6 x 10-5 uc/ml. On February 14, 1958, two samples of a milky white dispersion showed a concentration of suspended material amounting to 4700 uc/ml, and 419 uuc/ml. On June 24, 1959, a sample containing soluble effluent revealed no activity in excess of instrument background while another sample (milky white dispersion) showed a concentration of 3880 uuc/ml. On January 21, 1959, another dispersion sample showed a concentration of 3300 uuc/ml. On November 10, 1959, an undissolved sample collected in the stream showed a concentration of 11,400 uuc/ml. On November 11,

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1959, another sample revealed a concentration of 5900 uuc/ml.
Russe stated that since December 14, 1959, several samples run by his office showed concentrations less than instrument background. Russe stated that "Mandle has taken to watch the pH control) and therefore has had a better control of effluent release to the storm sever".

#### 18. Posting and Labeling

After being querried as to the relative (cleanliness) of the radiation signs, Hubbard noted that he had posted various areas' inside and outside the plant with required caution signs and symbols just prior to the inspection. There were several areas in the plant and outplant in which radiation areas existed which were not posted. The areas in which over 30 odd druns of ThOH and about 30 druns of ThO2 were stored were not posted with a sign denoting a radiation area. Each of the aforementioned drums contained over 500 lbs. of material. According to Mandle, radiation measurements made with a calibrated GM or June showed levels from 7.5 mr/hr to 12 mr/hr at 1° from the drums. The area in which ever 30 drums of ThOR were stored outside the plant was not posted either with a caution, radiation material sign or the radiation area sign. The drums themselves were not labeled with a caution, radioactive material sign, amount, or type of material. Several hundred 120 lb. bags of monasite sand containing from 3 to 3.5% ThO, which were stored in the monasite storage area, were not labeled with any radioactivity sign or amount, or type of material. The storage area was properly posted with both a caution sign and symbol.

The piles of sludge such as the silica (mesothorium) and gray phosphate cake at which radiation measurements at 1° from the pile showed 11 and 15 mr/hr, respectively, were not posted with a radiation area or a caution, radioactive material sign. These piles contained approximately 200 tons of materials(4 tons ThO<sub>2</sub>). Other piles of sludge stored outplant which include waste treatment yttrium and reworked silica sludge were not posted with any caution, radioactive material sign or symbol. Hubbard stated that he had run out of signs and noted that he was aware that these areas required proper posting. The entrances to the production and waste treatment plants were noted to be properly posted.

#### 19. Incineration

Mandle stated that he periodically incinerated paper bags in which the monasite sand is shipped and wipes and wood contaminated with thorium. He added that he has not evaluated the hazard involved in the burning of these contaminated materials by taking air samples, soil samples, etc. during and after burning of the waste, respectively.

#### 20. Records

Records of procurement, receipt, transfer, film badge, physical exams, were found to be in order. No records were maintained on waste disposal. Direct radiation survey records did not include the type of instrument used and did not, in the March 27, 1959 survey (Exhibit C), record the measurements in mr/hr. The survey records shown in Exhibit B and C did not completely evaluate the hazard due to storage of piles of radioactive sludge outside the plant at which levels in excess of 10 mr/hr existed.

#### 21. Other Part 40 Licenses

### A. License C-3623

Under License C-3623, a 50 lb. drum of uranium-magnesium acrap was procured by R. Mandle for use in experimental work relating to the recovery of uranium from magnesium fluoride scrap. No work was ever performed according to Mandle. The 50 lbs. of material which was reportedly posted was stored in the company storage warshouse. Records of receipt were available.

#### B. License R-132

According to Mandle, no work is in progress under license R-132, Davison's Pompton Plains or Curtiss Bay plants.

All work at Pompton Plains is being performed under license R-196. No work under R-132 or R-196, according to Mandle, is being performed at the Crutiss Bay, Maryland, plant of Davison Chemical Co. Under R-132, Mandle stated that 100 lbs. of thorium bomb reduction residues had been transferred by him to the Davison Chemical Co. plant at Erwin, Tennessee.

PURCHASE

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DAVISON CHEMICAL COMPANY

Propress through Chemistry

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NO NO 2551

DATE 3-25-59

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## EXHIBIT B .

January 6, 1959

R. M. Mandle

FILES

Survey of Plant for St. John I-Ray Lab

Monazite Storage area Ball Mill area Monasite transfer drums Centrifuge and Press #5 Barrels stored by tank #31 Crystal Dissolve Tank	2-10 mr/hr 1 mr/hr 3 mr/hr 1-2 mr/hr 1-2 mr/hr 1 mr/hr
Tank #1 Tank #2 Tank #3 Tank #4-5 Packing room	1 mr/hr 1 mr/hr 0.5-1mr/hr 0.1-0.2 mr/hr 0.2-0.3 mr/hr

Dr. Isenberger - Califon 49

Badges - 150 for \$85.00 - Send holders and film. Enter numbers on reports and return them to St. John. They process and notify. We keep film and reports.

New AEC regulations require a 13 week accumulation -Mr. Isenberger suggests we purchase and load two films and keep one of them in for 13 week period.

Holders \$1.50 sach.

R.M.M.

MCB:1

## BIHIBIT C

## Survey of Plant (New batteries installed in Geiger Counter)

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