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OPTIONAL FORM NO. 10

UNITED STATES GOVERNMENT

## Memorandum

TO : W. B. McCool, Secretary  
THRU : H. L. Price, Acting Director of Regulation  
FROM : Robert Lovenstein  
Acting Director  
Division of Licensing and Regulation  
SUBJECT: PETITION FOR RULEMAKING -- 10 CFR <sup>b7</sup> LICENSING OF SOURCE MATERIAL  
SYMBOL: DLAR:LJ

DATE: March 15, 1961

Attached for your records and for reproduction for the information of the Commission is a communication dated March 6, 1961, from American Potash and Chemical Corporation (Lindsay Chemical Division). This communication constitutes a petition for rule-making and requests:

1. that Section 40.13(c) (1) of 10 CFR 40 be amended to re-establish an exemption from licensing for

"rare earth metals and compounds, mixtures and products containing not more than 0.25% by weight thorium, uranium, or any combination of these;

and

2. pending completion by the AEC of technical studies relating thereto and action by the Commission to re-establish this exemption on a permanent basis, that the Commission amend 10 CFR 40 on a temporary basis so that it will not be necessary for the Corporation to suspend a major portion of its rare earth chemicals business and cause a disruption in the business of its customers.

The Division of Licensing and Regulation will submit a report promptly to the Commission with recommendations as to the action the Division concludes is warranted under the circumstances. A copy of the petition is in the Public Document Room.

Enclosure:

Ltr dtd 3/6/61 fr American Potash  
& Chemical Corporation

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AMERICAN POTASH & CHEMICAL CORPORATION  
NINETY-NINE PARK AVENUE, NEW YORK 16, NEW YORK

March 6, 1961

Mr. H. L. Price  
Director  
Division of Licensing Regulation  
U. S. A. E. C.  
Washington 25, D. C.

Dear Mr. Price:

This letter is a request that Section 40.13 (c)(1)  
"Unimportant Quantities of Source Material" of the Regulation of  
the Commission 10 CFR Part 40, "Licensing of Source Material",  
be amended to read as follows:

"Any quantities of thorium contained in (i) incandescent gas  
mantles; (ii) vacuum tubes; (iii) welding rods; or (iv) rare  
earth metals and compounds, mixtures and products containing  
not more than 0.25% by weight thorium, uranium or any  
combination of these."

thereby restoring the exemption applicable prior to February 13,  
1961 to such rare earth materials.

Attached hereto is our application for this amendment which  
contains evidence of the irreparable damage which will be done to the  
rare earth industry by not restoring the long-standing exemption for  
rare earth materials having a thorium plus uranium content of less  
than 0.25% by weight. This application also shows that this  
damage is needless because there is no health hazard involved in  
the use, possession or handling of rare earth materials having a  
thorium content of less than 0.25% by weight. We are fully  
familiar with the rare earth business in the United States; we  
know of no health problems caused by the handling of these materials  
from 1946 to 1961 during which period the exemption was in the  
Commission's regulations. We know of no health problems even before  
1946.

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We conclude and show the following:

1. The thorium content limit of 0.05% is unnecessary from health - physics considerations.
2. The thorium content limit of 0.05% is unrealistic and impractical from a technical and analytical standpoint.
3. Licensing procedures called for under these regulations will do irreparable damage to the present and future rare earth industry by removing the rare earths from commercial application due to the requirements of Part 20. Increased capital costs, added professional fees, lengthy and costly labor negotiations, administrative burdens and probable higher wage rates will remove rare earth materials from practical commercial consideration. The result will be to cripple the rare earth industry and cause considerable unemployment.
4. A large number of consumers of rare earth materials will be subjected to licensing and regulation needlessly.
5. The licensing program and the burden on the Commission of administering it will be significantly increased.

Many of our present customers will have received within the next one to five weeks the amount of contained thorium in rare earth materials permissible under general licensing. Unless they have secured specific licenses in the meantime, we will be prohibited from shipping rare earths to them. We will also be prohibited from shipping single orders for rare earths where the contained thorium limits for single shipments are exceeded. The effect will be to suspend a major portion of our business and the business of our customers.

Since time is of the essence and any delay, potential or real, in the permanent restoration of the original exemption would cause us to suspend shipments of our products which are vital raw materials to many consumers in important industries, specific licensing of these consumers under Part 40 might be required

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almost immediately. This action would subject them to the requirements of Part 20. To avoid the Part 20 requirements, many of these consumers will suspend operations or use competing non-rare earth materials where feasible. This will prematurely and perhaps permanently cripple the rare earth industry even though the new regulations are effective only for the time required to effect the permanent restoration of the original exemption.

Therefore, inasmuch as we will be forced by the new regulations within several days to cease shipments to, and disrupt the business of, a number of our long - standing and new customers, we urgently request prompt action on a temporary amendment re-instituting the exemption for rare earth materials contained in the regulations from 1946 to 1961, so that business may continue, such action being followed as soon as possible by permanent restoration of the original exemption.

If you should require further information or should you desire further discussion of this subject, please contact the writer at the above address.

Very truly yours,  
AMERICAN POTASH & CHEMICAL CORP.

By:                     /s/                      
Paul E. McCoy  
Manager  
Lindsay Chemical Sales

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APPLICATION FOR AMENDMENT OF PART 40  
REGULATIONS OF THE ATOMIC ENERGY COMMISSION  
RE: THORIUM CONTENT OF RARE EARTH MATERIALS

American Potash & Chemical Corporation

March 6, 1961

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PART I

SUMMARY STATEMENT AND CONCLUSIONS

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Part I

Summary Statement And Conclusions

10 CFR, Part 40 ("Control of Source Material"), Chapter I, Title 10 defines source material as:

1. Uranium or thorium or any combination thereof, in any physical or chemical form or,
2. Ores which contain by weight one-twentieth of one per cent (0.05%) or more of (i) uranium, (ii) thorium or (iii) any combination thereof.

A chemical mixture, compound, solution or alloy in which the source material is by weight less than 0.05% of the mixture, compound, solution or alloy is exempt from the regulation

Under these regulations, a general license is issued to domestic commercial and industrial firms, and research, educational and medical institutions for research, development, educational or commercial purposes authorizing the use and the transfer of not more than fifteen (15) pounds of source material at any one time, provided that no such person shall receive more than a total of one hundred fifty (150) pounds of source material in any calendar year.

We have concluded and propose to show that under these regulations:

1. The thorium content limit of 0.05% is unnecessary from health-physics considerations.
2. The thorium content limit of 0.05% is unrealistic and impractical from a technical and analytical standpoint.
3. Licensing procedures called for under these regulations will do irreparable damage to the present rare earth industry to the extent that considerable unemployment may be involved.
4. A large number of consumers of rare earth materials will be subjected to licensing and regulation needlessly.



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Summary Statement & Conclusions

5. The licensing program and the burden on the Commission of administering it will be significantly increased.

After lengthy study of the problem and from our long experience in producing and handling thorium and rare earth materials, we urge that:

Section 40.13 (c)(1) of Part 40 of the Commission's regulations, as published in the Federal Register of January 14, 1961, be amended to read as follows:

"Any quantity of thorium contained in (i) incandescent gas mantles; (ii) vacuum tubes; (iii) welding rods; or (iv) rare earth metals and compounds, mixtures and products containing not more than 0.25% by weight thorium, uranium or any combination of these."

The effect of the proposed amendment is to add to the three exemptions presently listed in the clause quoted the exemption for rare earth materials containing less than 0.25% by weight thorium, restoring an exemption which was in the regulations from 1946 to 1961.

It was in consideration of the foregoing that the letters of October 26, 1960 and January 30, 1961 were submitted to the Commission by Mr. C. R. Lindsay, III, Vice President, American Potash & Chemical Corporation.

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PART II

RARE EARTH MANUFACTURING EXPERIENCE OF APPLICANT  
AMERICAN POTASH & CHEMICAL CORPORATION

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Part II

Rare Earth Manufacturing Experience of Applicant  
American Potash & Chemical Corporation

American Potash & Chemical Corporation is a corporation organized in 1926 under the laws of the state of Delaware and consolidated in the same year with the American Trona Corporation, a Delaware corporation organized in 1913. It is engaged in the production, manufacture and distribution of a wide variety of chemicals which are sold throughout the United States and in many foreign countries. These chemicals include boron chemicals, lithium chemicals and lithium ores, chlorates and perchlorates, manganese dioxide, sodium chemicals, potassium chemicals, agricultural chemicals, thorium, yttrium and rare earth chemicals and additional specialty chemicals including those of rubidium and cesium. Plants are located at Trona, California, Henderson, Nevada, Los Angeles, California, Aberdeen, Mississippi and West Chicago, Illinois. Its National Northern Division, carrying on research and testing in the ordnance and explosives fields, is located at West Hanover, Massachusetts. Manufacturing facilities of American Lithium Chemicals, Inc. and San Antonio Chemicals, Inc., in each of which American Potash & Chemical Corporation holds a majority interest are located at San Antonio, Texas.

During 1960 the net sales of American Potash & Chemical Corporation totalled \$50,546,003. Thorium, yttrium and rare earth chemicals is one of the Company's main product lines. The Company and its domestic subsidiaries employ approximately 2,000 people. A large number of people are employed in the Company's plant at West Chicago, Illinois in the production and distribution of thorium, yttrium and rare earth chemicals.

The Company's West Chicago, Illinois plant was a result of its acquisition of Lindsay Chemical Company in May of 1958. Lindsay was founded in 1902 as Lindsay & Company and was incorporated in Illinois in 1904. It is presently the largest refiner in the United States of thorium, rare earth chemicals and

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Rare Earth Manufacturing Experience of Applicant  
American Potash & Chemical Corporation

and yttrium. Lindsay originally manufactured incandescent mantles, which required thorium nitrate as one of the raw materials. Previous to World War I thorium nitrate used for mantles was imported from Germany in a refined state. When that war eliminated this source, Lindsay commenced and ever since has continued its own processing of monazite sand from which thorium, the rare earths and yttrium are derived. As the demand for mantles declined and with it the demand for thorium, Lindsay supplanted that business by developing refining processes for the rare earth chemicals as new uses were discovered for these elements. The mantle business was sold as of December 31, 1953. With the advent of the atomic energy program, a new demand for thorium nitrate came into being. At the present time Lindsay's business is the production, manufacture and sale of thorium, rare earth and yttrium chemicals.

Lindsay played a significant role in furnishing thorium and rare earth products to the Manhattan Project in connection with the development of the atomic bomb during World War II. Since 1945 Lindsay has furnished the bulk of its thorium production to the Government.

The rare earths consist of a group of fifteen elements which are always found together in nature. The principal source is monazite sand, which as noted above, Lindsay has processed since World War I. Another source is bastnaesite. Lindsay has processed bastnaesite but is not currently doing so. Significantly important commercial areas of application for rare earth chemicals include the manufacture of arc carbons, lighter flints, metallurgical additives, glass, ceramics, precision lenses, television tubes, electronic ceramics and microwave devices.

Lindsay was one of the first to develop, and believes it is the leader in, the commercial application of the ion exchange process to the separation of highest purity yttrium and rare earth products. Among the potential future uses

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Rare Earth Manufacturing Experience of Applicant  
American Potash & Chemical Corporation

of these elements is the application of samarium, gadolinium and europium as neutron absorbers in the control rods of nuclear reactors. Other separated rare earths, such as dysprosium and erbium may find a use as an addition to these rods to balance neutron absorption.

Thorium is the only material known, except for uranium, which, though not itself fissionable, can be converted to fissionable material. This is done by placing it in an atomic "converter" or "breeder" reactor. In 1952 arrangements were made with the Atomic Energy Commission for Lindsay to expand its production of thorium chemicals and as a result a large addition to its facilities was completed in July, 1954. This expansion was designed to facilitate the production of thorium on a basis which would also provide practical commercial routes for the processing of rare earth chemicals. The acceptability of this process was based upon the regulations then in effect, namely the Code of Federal Regulations, Title 10, Part 40, as amended January 1, 1954. This regulation listed the following exemption:

P 40.60 Schedule I

"(f) Rare earth metals and compounds, mixtures and products containing not more than 0.25% by weight thorium, uranium, or any combination of these."

Lindsay entered into a contract with the Commission for the sale of thorium products, which contract expired December 31, 1956. It operated on a new contract running until the end of 1959 under which the Government acquired thorium salts. In addition to this outlet for its thorium products, the Corporation produces thorium compounds for use in a number of other applications including thorium-magnesium alloys for jet engines and missiles, thorium salts for nuclear fuels, thoriated tungsten wire for electronic tubes, refractories for high temperature purposes, catalytic agents for various industries. high

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Rare Earth Manufacturing Experience of Applicant  
American Potash & Chemical Corporation

quality optical glass, incandescent mantles, and various chemical reagents.

Of the total thorium, rare earth and yttrium materials produced and sold by the Corporation's West Chicago, Illinois plant, the rare earth chemicals and polishing compounds represent the major portion.

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PART III

RADIATION EXPERIENCE AT WEST CHICAGO PLANT IN HANDLING  
RARE EARTH AND YTTRIUM PRODUCTS

The data presented here show that both the air-borne alpha activity and gamma radiation levels encountered in the manufacture and handling of rare earth materials containing on the order of 0.25% thorium are quite small, do not present a hazard, and do not necessitate radiation monitoring.

Contents

1. The problem and its significance.
2. Scope of data.
3. Method of determination of radiation exposure.
4. Explanation of units.
5. Facilities and products covered.
6. Radiation data.

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### 1. The Problem and its Significance

On the effective date of 10 CFR Part 40 published in 26 FR 284, January 14, 1961, manufacturers and users of rare earth materials are required to operate under a tighter specification for the thorium content of these materials if rare earth materials are to be exempt from specific licensing. In effect, the maximum exempted content of thorium was reduced from 0.25% to 0.05% in the revised Part 40 rules.

The uranium content of rare earth materials is insignificant since the starting ores (monazite, etc.) contain only small amounts of uranium. Uranium contents for rare earth materials are in the parts-per-million range. Therefore this problem is concerned essentially with thorium contamination.

The data presented here show that both the air-borne alpha activity and gamma radiation levels encountered in the manufacture and handling of rare earth materials containing on the order of 0.25% thorium are quite small, do not present a hazard, and do not necessitate radiation monitoring.

The information herein presented was summarized from the records maintained at the West Chicago plant by Dr. R. M. Healy, Radiation Safety Officer at the West Chicago plant, with the assistance of Dr. H. E. Kremers.

### 2. Scope of Data

In 1956 the West Chicago plant, then known as The Lindsay Chemical Company, initiated a broad and intensive radiation safety program. The program was activated in anticipation of the impending rules to be announced in 10 CFR Part 20 ("Standards for Protection Against Radiation", January 29, 1957). This program has been sustained without interruption. It has been pursued with the vigor necessary to be in compliance as lower maximum radiation limits have been announced.

The program includes film badge service, and air concentration measurements. All operating personnel, and in the thorium plant foremen also, are required to wear badges whenever they are at work. Rules for the wearing of film badges are vigorously enforced. Many of these people transfer back and forth between the thorium and rare earth sections of the plant. While many of the jobs would not require a badge, there is a considerable mixing of people and jobs. The decision to badge all operating personnel was made to maintain flexibility.

The air sampling program has also been unusually thorough. Over seven thousand air samples have been taken since 1956. The data presented here of course do not reflect the full magnitude of the air sampling program. Experience quickly showed that it was not necessary to sample the areas of present interest extensively, due to the low radiation levels encountered. The main effort has been directed toward operations involving handling of thorium materials.

The Radiation Safety Program is under the direction of the Radiation Safety Officer. Dr. H. E. Kremers formerly held this position. It is presently the responsibility of Dr. R. M. Healy. A full time technician is employed to take samples and run the film badge program. A radiation specialist, Dr. R. S. Landauer, is retained as consultant.



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3. Method of Determination of Radiation Exposure

Film badges are supplied and processed by the E. S. Landauer, Jr. and Company, 3920 - 216th Street, Matteson, Illinois.

The West Chicago plant possesses two alpha scintillation detectors, with all necessary auxiliary equipment for obtaining and measuring air concentrations.

The method followed is that used by the Health and Safety Division, New York Operations Safety Office when its personnel made a survey of the thorium plant in 1956. A known quantity of air is drawn through a piece of Whatman 41 filter paper. A standard sampling time of fifteen minutes is used in this plant. The samples are stored one week in order to improve the reproducibility of the results. This recommended technique was necessary in the thorium plant, but would have little effect in those areas where materials are being mechanically processed. Samples were ordinarily counted for five minutes, using generally accepted techniques for calibration and background correction.

4. Explanation of Units

For air-borne thorium measurements,

Let

$l/m$  = liters per minute air flow in sampler

$m$  = minutes sampling time

$c/m$  = alpha counts per minute on paper sample

$d/c$  = alpha disintegrations per count (determined frequently from standard alpha source)

$d/m$  = alpha disintegrations per minute

Microcuries of thorium =  $\mu cTh = \frac{d/m}{2.2 \times 10^6 d/m/\mu c}$

ML = maximum permissible level

then

$$\mu cTh/ml = \frac{(c/m)(d/c)}{2.2 \times 10^6 (l/m)(m)(1000 ml/l)}$$

$$\% ML = \frac{(\mu cTh/ml)(10^{11})(100)}{5}$$

Film badge readings are reported as millirems per year as reported to us by the film badge processor.

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### 5. Facilities and Products Covered

The West Chicago plant has approximately 266,000 square feet; approximately 80,000 square feet is used for thorium processing and about 75,000 square feet for rare earth processing. Ore and rare earth raw material storage covers 24,000 square feet, and the balance is used for offices, services, maintenance facilities, research laboratories, and product warehousing.

Processing starts with monazite ore, a phosphate of rare earths and thorium, and consists of the following basic steps which are also summarized on the accompanying flow chart.

- (a) Opening the ore by a wet chemical extraction process.
- (b) Preliminary chemical separation of thorium from rare earths, the thorium and rare earth intermediates are processed separately to remove additional rare earths and thorium.
- (c) Recovery of rare earths as rare earth sodium sulfate for storage or conversion to other rare earth compounds.
- (d) Separation of the rare earth mixture into cerium chemicals and didymium materials, and further separation of didymium into other individual rare earth products.

The input of rare earths into the West Chicago plant, in the form of rare earth sodium sulfate containing 42% rare earth oxide, is several million pounds per year. The plant produces for sale a nearly corresponding quantity of all rare earth products, this quantity varying somewhat with the type of rare earth product and market conditions. Most of the production is in the form of rare earth and commercial cerium products such as rare earth chloride Code 340, rare earth oxide Code 330, rare earth fluoride Code 370, cerium hydrate Code 201, and various rare earth and cerium-based glass polishing compounds. Production and sale of high purity, individual separated rare earth materials is relatively small compared to the commercial-type rare earth products.

Radiation data are presented in Section 6 of this document for operations involving the handling, preparation, and packaging of the following types of rare earth materials.

- (a) Glass Polishes. These are essentially rare earth or cerium oxides specially prepared for glass and optical polishing. BARNESITE is essentially a rare earth oxide, and CEROX is a moderately pure cerium oxide. Processing involves wet chemical and slurry treatments in open tanks, filtration, calcining to oxide in continuous and batch furnaces, ball milling, and dry screening and blending operations. Dusting is apt to occur in the calcining and screening steps, and is of considerably greater magnitude than that found in glass polishing operations where wet slurries are universally handled.
- (b) Cerium Hydrate Manufacture. This operation includes wet chemical separation of cerium from the rare earth mixture, filtration, drying, pulverizing and packaging operations. The last three steps involve the handling

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ing of dusty materials. Products are cerium hydrate and didymium carbonate.

- (c) Rare Earth Oxide is prepared by calcining rare earth oxalate or rare earth carbonate in continuous, rotary furnaces. The product is a powder largely in the -200 mesh range, and is susceptible to dusting.
- (d) High Purity Rare Earth and Yttrium Oxides are produced by calcining the rare earth oxalates obtained from ion exchange effluent solutions.
- (e) Rare Earth Sodium Sulfate Handling and Storage. This salt is precipitated from aqueous solution, recovered by filtration on a continuous filter, and the filter cake stored in bulk or further processed into rare earth chemicals.
- (f) Rare Earth Chloride is prepared from rare earth sodium sulfate by chemical conversion to give an aqueous solution of rare earth chloride. The solution is treated to remove deleterious impurities (including thorium), and the treated solution evaporated in open kettles to the proper composition. The evaporated, concentrated rare earth chloride liquor is allowed to solidify, and is broken up and packed for shipment.
- (g) Rare Earth Fluoride is prepared by precipitation from aqueous solution, filtered, and the filter cake dried. The dry product is pulverized to powder or converted to pellet form.

S. Radiation Data

Some considerations which help to evaluate the data given in the following tables are:

- (1) The numbers are biased on the high side to an unknown but probably significant degree due to proximity of thorium processing.
- (2) The uncertainty in any one air sample is relatively large. The counting uncertainty is relatively high since only a few counts per sample were recorded in these low level areas. This fact accounts for a substantial part of the variation in the measured air concentrations at different times. It will be recognized that the averaged concentrations are quite sufficiently precise for the present purposes.
- (3) Film badge radiation exposures are shown for the last two years only because for this period the processor's IBM machine totaled values are reported automatically. Monthly values from previous years are similar to

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monthly values for the years cited. The annual totals are obviously quite low, and can be obtained if necessary.

- (4) The through-put of West Chicago plant is many times that of any particular customer.

Table 1. A Record of Air Concentrations in the  
Rare Earth Processing Plant

| (a) <u>Glass Polishes</u>   | <u>Date</u> | <u>Sample No.</u> | <u>% ML</u> |
|---|-------------|-------------------|-------------|
| Air samples from areas where polishing materials are prepared, calcined, sifted, and packed. Samples 1783-1789 and 3404-3458 relate to handling of BARNESITE. | 2-12-58     | 1783              | 19          |
|   |             | 1784              | 39          |
|   |             | 1785              | 37          |
|   |             | 1786              | 18          |
|   |             | 1787              | 15          |
|   |             | 1788              | 21          |
|   |             | 1789              | 21          |
|   |             | Average           | 24.3        |
| Samples 1875-1879 and 7074-7091 are from areas where cerium oxide polishes (CEROX) are calcined.  | 2-27-58     | 1875              | 1           |
|   |             | 1878              | 1           |
|   |             | 1879              | 1           |
|   |             | Average           | 1           |
| Samples 7025-7031 are breathing zone samples from areas where cerium oxide polishes are dry screened and packed.  | 1-15-59     | 3451              | 13          |
|   |             | 3453              | 3           |
|   |             | 3456              | 1           |
|   |             | 3458              | 14          |
|   |             | Average           | 7.8         |
| Thorium contents:-<br>BARNESITE <0.05%<br>SUPER-CE 0.10<br>CEROX 0.18<br>Others 0.09-0.21   | 1-16-59     | 3404              | 14          |
|   |             | 3406              | 20          |
|   |             | 3407              | 0           |
|   |             | Average           | 11.3        |
|   | 10-20-60    | 7074              | 25          |
|   |             | 7076              | 21          |
|   |             | 7091              | 0           |
|   |             | Average           | 15.3        |
|   |             | 7020              | 7           |
|   | 10-20-60    | 7021              | 8           |
|   |             | 7022              | 13          |
|   |             | 7025              | 10          |
|   |             | 7028              | 28          |
|   |             | 7031              | 16          |
|   |             | Average           | 13.7        |
| Average of all samples  |             |                   | 14.1        |

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| (b) <u>Cerium Hydrate</u> | <u>Date</u>            | <u>Sample No.</u> | <u>% ML</u> |
|---------------------------|------------------------|-------------------|-------------|
| General air samples       | 2-27-58                | 1873              | 1           |
| in areas involving        |                        | 1874              | 3           |
| drying of rare earth      |                        | 1888              | 9           |
| hydrate in manually-      |                        | 1889              | 4           |
| loaded and unloaded       |                        | 1890              | 4           |
| tray driers, drying       |                        | 1891              | 3           |
| of cerium hydrate in      |                        | 1892              | 6           |
| manually-loaded and       |                        | 1893              | 8           |
| unloaded tray driers,     |                        | 1904              | 4           |
| and wet chemical          |                        |                   |             |
| separation of cerium      |                        | Average           | 4.7         |
| from the rare earth       | 2-29-58                | 1931              | 0           |
| mixture.                  |                        | 1940              | 1           |
|                           |                        | 1942              | 0           |
| Thorium content:- 0.18%   |                        | 1943              | 7           |
|                           |                        | 1945              | 2           |
|                           |                        | 1946              | 1           |
|                           |                        | Average           | 1.8         |
|                           | 10-20-60               | 7082              | 1           |
|                           |                        | 7085              | 0           |
|                           |                        | 7087              | 1           |
|                           |                        | 7089              | 0           |
|                           |                        | 7097              | 2           |
|                           |                        | 7116              | 10          |
|                           |                        | 7118              | 29          |
|                           |                        | 7119              | 32          |
|                           |                        | 7121              | 17          |
|                           |                        | 7122              | 17          |
|                           |                        | 7125              | 12          |
|                           |                        | 7128              | 21          |
|                           |                        | 7129              | 12          |
|                           |                        | 7130              | 7           |
|                           |                        | 7131              | 0           |
|                           |                        | 7133              | 4           |
|                           |                        | 7134              | 0           |
|                           |                        | 7136              | 20          |
|                           |                        | 7137              | 9           |
|                           |                        | Average           | 10.2        |
|                           | Average of all samples |                   | 7.3         |

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| (c) <u>Rare Earth Oxide</u>  | <u>Date</u> | <u>Sample No.</u> | <u>% ML</u> |
|--|-------------|-------------------|-------------|
| Samples 3546-3566 were taken inside the closed room in which rare earth oxide is calcined in unattended continuous rotary calciners. This operation is dusty. The values have not been weighted for personnel exposure time. | 1-27-59     | 3546              | 41          |
|  |             | 3548              | 38          |
|  |             | 3550              | 43          |
|  |             | 3552              | 20          |
|  |             | 3554              | 30          |
|  |             | 3561              | 28          |
|  |             | 3566              | 34          |
|  |             | Average           | 33          |
| Samples 7086-7109 are general air samples taken in the area close to the calciner where material is screened and packed.   | 10-20-60    | 7086              | 7           |
|  |             | 7098              | 4           |
|  |             | 7102              | 5           |
|  |             | 7104              | 30          |
|  |             | 7106              | 8           |
|  |             | 7109              | 34          |
|  |             | Average           | 15          |
| Thorium content:- 0.22%  |             |                   |             |
| Average of all samples   |             |                   | 24          |
|  |             |                   |             |
| (d) <u>Ion Exchange - Purified</u>   |             |                   |             |
| <u>Rare Earths</u>   | 2-18-57     | 408               | 10          |
|  | 3-12-57     | 449               | 27          |
| Thorium content of most products produced:- <0.05%   | 9-19-57     | 1006              | 7           |
|  |             | 1009              | 7           |
|  | 10-22-57    | 1141              | 28          |
|  | 1-29-58     | 1569              | 26          |
|  |             | 1570              | 27          |
|  |             | 1572              | 8           |
|  |             | 1574              | 1           |
|  |             | 1578              | 18          |
|  |             | 1580              | 20          |
|  |             | 1582              | 5           |
|  |             | 1585              | 29          |
|  |             | 1587              | 16          |
|  |             | 1589              | 27          |
|  |             | 1593              | 10          |
|  |             | 1595              | 16          |
|  |             | 1597              | 27          |
|  |             | 1600              | 10          |
|  |             | 1602              | 1           |
|  |             | 1603              | 3           |
|  |             | 1604              | 25          |
|  |             | Average           | 15.8        |

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| <u>Ion Exchange - Cont'd.</u> | <u>Date</u> | <u>Sample No.</u> | <u>% ML</u> |
|-------------------------------|-------------|-------------------|-------------|
|                               | 1-13-59     | 3301              | 5           |
|                               |             | 3302              | 5           |
|                               |             | 3303              | 9           |
|                               |             | 3306              | 5           |
|                               |             | 3307              | 7           |
|                               |             | 3308              | 5           |
|                               |             | 3309              | 24          |
|                               |             | 3310              | 0           |
|                               |             | 3312              | 7           |
|                               |             | 3313              | 17          |
|                               |             | Average           | 8.4         |
|                               | 4-5-59      | 3973              | 9           |
|                               |             | 3975              | 11          |
|                               |             | 3976              | 0           |
|                               |             | 3977              | 0           |
|                               |             | 3979              | 0           |
|                               |             | 3981              | 14          |
|                               |             | 3983              | 27          |
|                               |             | 3985              | 0           |
|                               |             | 3989              | 11          |
|                               |             | 3990              | 9           |
|                               |             | 3991              | 3           |
|                               |             | 3992              | 24          |
|                               |             | 3993              | 9           |
|                               |             | 3996              | 13          |
|                               |             | 3997              | 5           |
|                               |             | 3999              | 20          |
|                               |             | 4001              | 9           |
|                               |             | 4003              | 1           |
|                               |             | 4004              | 13          |
|                               |             | 4005              | 22          |
|                               |             | Average           | 10          |
|                               | 6-2-60      | 6323              | 0           |
|                               |             | 6324              | 0           |
|                               |             | 6325              | 2           |
|                               |             | 6326              | 2           |
|                               |             | 6327              | 0           |
|                               |             | 6328              | 5           |
|                               |             | 6329              | 21          |
|                               |             | 6330              | 47          |
|                               |             | 6331              | 12          |
|                               |             | 6332              | 6           |
|                               |             | 6333              | 15          |
|                               |             | 6334              | 0           |
|                               |             | 6336              | 0           |
|                               |             | 6337              | 0           |
|                               |             | 6338              | 20          |
|                               |             | 6339              | 0           |
|                               |             | 6340              | 6           |

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| <u>Ion Exchange - Cont'd.</u>        | <u>Date</u> | <u>Sample No.</u>   | <u>% ML</u> |
|--------------------------------------|-------------|---------------------|-------------|
|                                      |             | 6341                | 6           |
|                                      |             | 6342                | 2           |
|                                      |             | 6343                | 2           |
|                                      |             | 6344                | 71          |
|                                      |             | 6345                | 41          |
|                                      |             | 6346                | 9           |
|                                      |             | 6347                | 14          |
|                                      |             | 6348                | 2           |
|                                      |             | 6349                | 0           |
|                                      |             | 6350                | 14          |
|                                      |             | 6351                | 0           |
|                                      |             | 6352                | 0           |
|                                      |             | 6354                | 0           |
|                                      |             | 6355                | 0           |
|                                      |             | 6356                | 5           |
|                                      |             | 6357                | 12          |
|                                      |             | 6358                | 0           |
|                                      |             | 6359                | 3           |
|                                      |             | Average             | 9.1         |
|                                      |             | Average all samples | 10.9        |
| (e) <u>Rare Earth Sodium Sulfate</u> | 10-4-57     | 1005                | 1           |
|                                      | 1-28-58     | 1542                | 0           |
| General air sample of areas          |             | 1543                | 0           |
| where rare earth sodium              |             | 1544                | 0           |
| sulfate filter cake is dried         |             | 1545                | 0           |
| in rotary drier and handled          |             | 1546                | 0           |
| in bulk.                             |             | 1547                | 0           |
|                                      |             | 1548                | 0           |
| Thorium content:- 0.11%              |             | Average             | 0           |
| (f) <u>Rare Earth Chloride</u>       | 1-28-58     | 1555                | 12          |
|                                      |             | 1556                | 0           |
| Thorium content:- <0.05%             |             | 1557                | 3           |
|                                      |             | 1558                | 7           |
|                                      |             | 1559                | 3           |
|                                      |             | 1560                | 5           |
|                                      |             | Average             | 5           |
|                                      | 4-5-59      | 3958                | 0           |
|                                      |             | 3960                | 0           |
|                                      |             | 3962                | 0           |
|                                      |             | 3963                | 10          |
|                                      |             | 3964                | 10          |
|                                      |             | 3965                | 0           |
|                                      |             | 3966                | 0           |
|                                      |             | 3967                | 0           |
|                                      |             | 3974                | 3           |
|                                      |             | Average             | 2.6         |



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| <u>Rare Earth Chloride - Cont'd.</u> | <u>Date</u> | <u>Sample No.</u>   | <u>% ML</u> |
|--------------------------------------|-------------|---------------------|-------------|
|                                      | 6-2-60      | 6364                | 0           |
|                                      |             | 6365                | 6           |
|                                      |             | 6366                | 9           |
|                                      |             | 6367                | 0           |
|                                      |             | 6368                | 0           |
|                                      |             | 6369                | 0           |
|                                      |             | 6400                | 0           |
|                                      |             | 6402                | <u>16</u>   |
|                                      |             | Average             | 3.9         |
|                                      |             | Average all samples | 3.5         |
| (g) <u>Rare Earth Fluoride</u>       | 2-29-58     | 1931                | 0           |
|                                      |             | 1940                | 1           |
| General a'r samples in               |             | 1942                | 0           |
| rare earth fluoride                  |             | 1943                | 7           |
| preparation, drying,                 |             | 1945                | 2           |
| pulverizing, and pack-               |             | 1946                | <u>1</u>    |
| ing areas.                           |             | Average             | 1.8         |
| Thorium content: 0.22%               | 10-20-60    | 7101                | 15          |
|                                      |             | 7105                | 21          |
|                                      |             | 7107                | 10          |
|                                      |             | 7108                | 26          |
|                                      |             | 7109                | 26          |
|                                      |             | 7111                | 15          |
|                                      |             | 7112                | 20          |
|                                      |             | 7114                | 32          |
|                                      |             | 7115                | 10          |
|                                      |             | 7117                | 0           |
|                                      |             | 7120                | 36          |
|                                      |             | 7123                | 13          |
|                                      |             | 7126                | 9           |
|                                      |             | 7127                | 0           |
|                                      |             | 7129                | 3           |
|                                      |             | 7132                | <u>7</u>    |
|                                      |             | Average             | 15.2        |
|                                      |             | Average all samples | 11.5        |

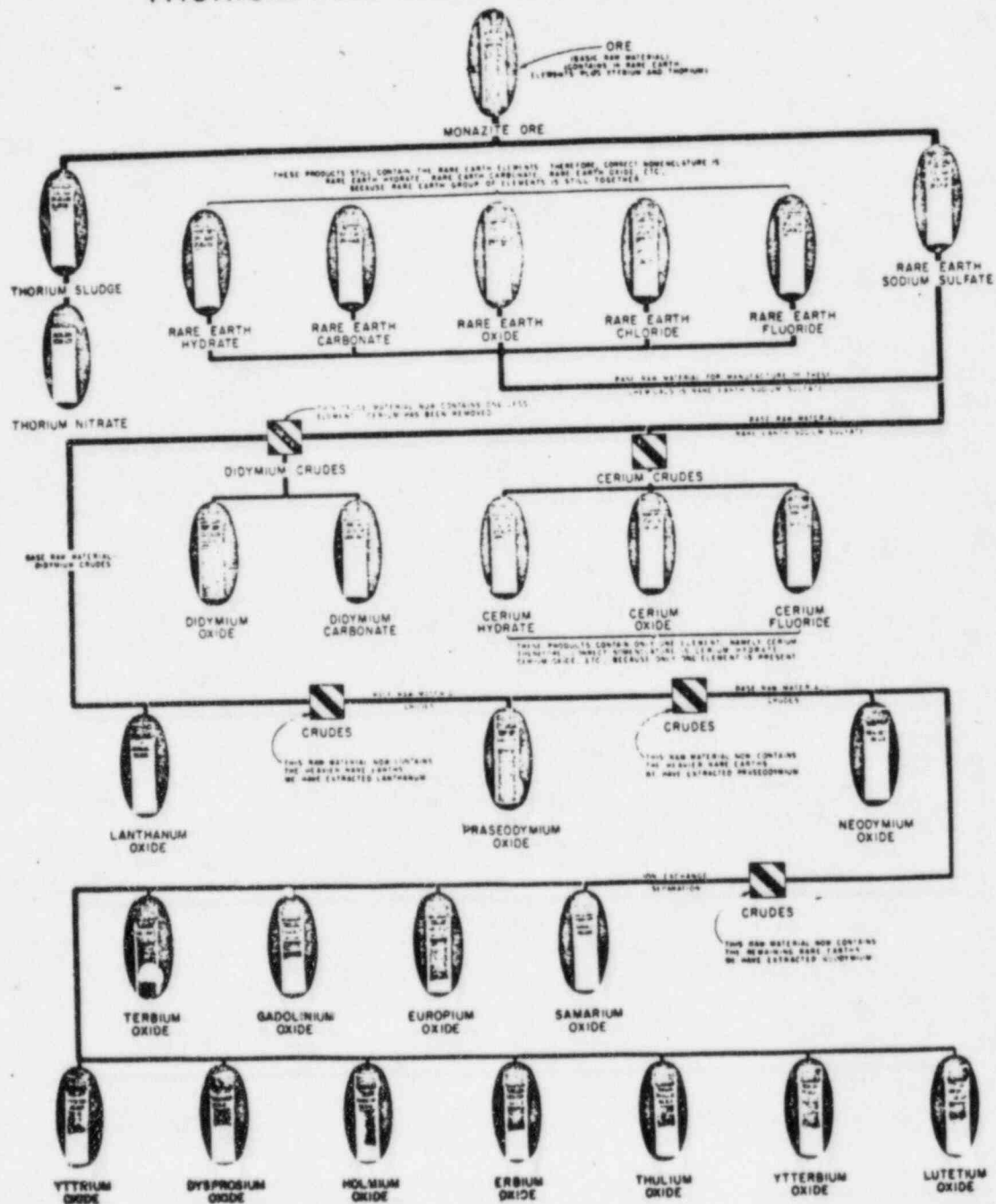
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Table II. Total Annual Exposures for Personnel  
Working with Crude Rare Earth Products,  
and with Purified Rare Earths in Ion Exchange Columns

| Badge No. | Job of Operator              | Year | Total Annual Exposure - Beta, Gamma - Millirems |
|-----------|------------------------------|------|---|
| 124       | Rare Earth Hydrate Operator  | 1960 | 455   |
|           |                              | 1959 | 210   |
| 67        | Fluoride Dryer-Grinder       | 1960 | 660   |
|           |                              | 1959 | 205   |
| 163       | Cerium Helper                | 1960 | 760   |
|           |                              | 1959 | 500   |
| 101       | Grinder Operator             | 1960 | 580   |
|           |                              | 1959 | 225   |
| 103       | BARNESITE Operator           | 1960 | 535   |
|           |                              | 1959 | 125   |
| 106       | Cerium Operator              | 1960 | 930   |
|           |                              | 1959 | 600   |
| 121       | Cerium Sifter                | 1960 | 135   |
|           |                              | 1959 | 60  |
| 117       | Rare Earth Fluoride Operator | 1960 | 265   |
|           |                              | 1959 | 455   |
| 136       | Rare Earth Hydrate Dryer     | 1960 | 580   |
|           |                              | 1959 | 160   |
| 126       | Ion Exchange Operator        | 1960 | 200   |
|           |                              | 1959 | 605   |
| 150       | Ion Exchange Operator        | 1960 | 660   |
|           |                              | 1959 | 210   |
| 100       | Ion Exchange Operator        | 1960 | 815   |
|           |                              | 1959 | 465   |
| 153       | Ion Exchange Operator        | 1960 | 1155  |
|           |                              | 1959 | 665   |
| 285       | Ion Exchange Operator        | 1960 | 785   |
|           |                              | 1959 | 870   |

**LINDSAY CHEMICAL DIVISION**  
*American Potash & Chemical Corporation*  
WEST CHICAGO, ILLINOIS

FLOW SCHEME  
THORIUM AND RARE EARTH CHEMICALS



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PART IV

ANALYSIS FOR THORIUM IN RARE EARTH MATERIALS

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#### Part IV

#### Analysis For Thorium In Rare Earth Materials

Determination of the thorium content of rare earth materials at levels below 0.25% thorium is so uncertain that the administration of the regulations with respect to permissible quantities for use and possession will be unrealistically impractical.

The analysis for thorium in materials containing rare earth has always presented problems. Although many years of experience by highly qualified chemists and analysts at the West Chicago plant have been accumulated in thorium assays, it is still felt that all practical methods for the analysis for thorium in rare earth materials are subject to more than ordinary uncertainty.

The degree of doubt in the accuracy of thorium determinations increases markedly as the thorium content decreases. Even relatively high thorium concentrations in rare earth materials (over one per cent thorium) are not easily determined accurately, and variations between duplicate samples normally are about ten to twenty per cent near the 1% thorium level. Variations between analysts working on identical materials often are also of this same magnitude. At low thorium concentrations (below one percent thorium), the reproducibility and precision of analyses become much poorer, and variations between assays of up to 100 per cent are common. At thorium concentrations near the 0.05% level in rare earth materials, determination of the thorium content cannot be done with reliable accuracy by practical methods adaptable to process and quality control. Reliable distinction between materials containing just below and just above 0.05% thorium is virtually impossible.

At concentrations up to about one-half per cent thorium, no good reliable methods exist for rare earth materials. Thorium assays at this level are more like "best estimates" than determinable numbers. The reliability of thorium determinations at the 0.25% thorium level in rare earth materials is

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Analysis for Thorium in Rare Earth Materials

considerably greater than reliability at the 0.05% level. Although not good at 0.25% thorium content, accuracy reliability at this level can be used for process and quality control, whereas reliability at the 0.05% level is so poor that in many cases, more credence must be put in the chemistry of thorium than in actual analytical results in order to evaluate thorium determinations.

Prior to 1945, the classical thorium pyrophosphate precipitation method was considered to be standard. Refinements in the thorium iodate precipitation method were made in 1945 and 1946, leading to acceptance of this method. In 1948, further research showed the iodate method to be inadequate for both high and low thorium concentrations, and precipitation of thorium with hexamethylenetetramine was established as the best method. This method is still considered to be the best available, but recent work has indicated that it too is subject to unreliability, particularly where the thorium content is low. Refinements in analytical techniques are used where possible; these include mesityl oxide liquid/liquid extraction, and colorimetric determination in small amounts of thorium. X-ray spectroscopy has some use where high thorium contents are encountered, but it is useless for low thorium concentrations.

Interfering elements in thorium analysis are, to mention a few, titanium, zirconium, and rare earths as well as fluorine and phosphorous. All of these are present either in the raw materials used for rare earth chemical manufacture, or in rare earth intermediate and many commercial rare earth products.

The analytical problems surrounding thorium are not unique to our operations. Among experienced analysts, it is well known that accurate assays for thorium are indeed difficult. Accuracies at low thorium levels in rare earth materials are more to be hoped for than to be realized.

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PART V

EFFECT OF REGULATIONS ON COMMERCIAL APPLICATIONS  
AND MARKETS FOR RARE EARTH MATERIALS

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Part V

Effect of Regulations on Commercial Applications  
& Markets for Rare Earth Materials

The arbitrary imposition of unrealistic limits on the thorium content of exempt rare earth materials, unnecessary for health-physics reasons, will cripple the rare earth industry and adversely affect employment and the economy generally. It will also add to the administrative burdens of the Commission.

The commercial rare earth market (excluding thorium materials) presently totals many millions of pounds annually with accompanying dollar volume. This volume is composed chiefly of approximately twenty "mixed rare earth" chemicals, rather than the separated rare earth elements, the market for which is only a small portion of the total. In addition to the mixed rare earth chemical category are approximately twelve rare earth glass polishing compounds for which the market is commercially considerable. These "commercial" rare earth materials are shipped in truckload quantities, and because of the thorium content of each material, many such shipments would exceed the "small quantity" requisites for general licensing. In this event, approximately fifteen to twenty commercial rare earth materials going to twenty-five to thirty-five industrial consumers in approximately seven different industries will be affected by the new regulations.

The large volume commercial consumption of rare earth materials has been established for approximately thirty-five years and involves such industries as arc carbon, glass and ceramics, metallurgical, electronic-ceramics and chemical. Under static conditions and, assuming no further growth in the use of rare earth materials by these industries, a minimum total of approximately twenty-five additional licenses must be issued in order to continue the use of rare earth materials under the present regulations. If the market growth and development of new uses proceeds in accordance with our estimates



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Effect of Regulations on Commercial Applications  
& Markets for Rare Earth Materials

if ever, before the rare earths could re-establish themselves in their presently important commercial applications. This would, of course, also result in the disappearance of the development of new uses and new markets for rare earth materials, a program which is essential to the survival of the rare earth industry. A delay in this program alone would in itself seriously damage the rare earth industry and cause considerable unemployment.

Since we have demonstrated that there is no reason from the health-physics point of view for not exempting from regulation rare earth materials having a thorium content of less than 0.25% by weight, that the technique of determining lesser content is so uncertain as to render administration of the regulations impractical, and that the failure to continue the long-existing exemption will seriously damage the industry and the economy, we urge that the regulations be amended as set forth above to restore the exemption as it existed prior to February 13, 1961.

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Effect of Regulations on Commercial Applications  
& Markets for Rare Earth Materials

and, we have good reason to believe they will, an additional four areas of industry will be involved, with the necessity for an approximate additional twenty-five to fifty licenses.

Rare earth chemicals must compete in most of their large volume commercial applications with non-rare earth materials. Competition on a cost-per-pound basis from non-rare earth materials is severe and any increase in the cost of using rare earths will naturally reduce the justification for their use and their economic advantages over other materials. Where costs of rare earth and non-rare earth materials are competitive, any inconvenience or added administrative burden incurred through the use of rare earth materials will cause the consumer to favor the non-rare earth competing product. An effect such as this will be the result if the rare earth industry is to operate under the present subject regulations.

In preparation of this application we reviewed this subject with some of our customers. We have every reason to believe that specific licensing under Part 40 and subjection to Part 20 would cause our present and potential customers to eliminate their present and future use of rare earth materials wherever feasible. Their objections were based upon increased capital costs, additional professional fees, lengthy and costly labor negotiations and probable higher labor rates as a result of the requirements of Part 20. Therefore, we are convinced that the effect of specific licensing for rare earth materials containing less than 0.25% thorium will be to cause present and potential users of these materials to seek competitive substitutes which are not so regulated, wherever and whenever possible.

The successful substitution of non-rare earth materials in applications where rare earths are important would seriously damage the commercial rare earth industry. Considerable unemployment would occur. It would then be some time,