8 pgs

LJOH 70-36

ESTABLISHED 1867

MALLINCKRODT CHEMICAL WORKS.

MANUFACTURERS OF
FINE CHEMICALS FOR MEDICINAL, PHOTOGRAPHIC
ANALYTICAL AND INDUSTRIAL PURPOSES

FACTORIES, ST. LOUIS, JERSEY CITY, MONTREAL
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CODES
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BENTLEY'S COMPLETE PHRASE
BENTLEY'S SECOND PHRASE

SECOND AND MALLINCKRODT STS ST. LOUIS, 7, MO.

Mallinckrodt FINE CHEMICALS Standard Since 1867

4 February 1958

Mr. Lyall Johnson
Licensing Division
U. S. Atomic Energy Commission
Washington, D.C.

SUBJECT: Special Nuclear Materials License No. SNM-33

Dear Mr. Johnson:

Some of our customers for our UO_2 of up to 5% enrichment have indicated a preference for blended lots in excess of the "limited safe" batch size. In order to provide such material, it is necessary to utilize blending equipment.

We have designed a blending facility which we believe quite adequate from the standpoint of criticality control and dust control and are requesting that our license be modified to include this blending facility as a part thereof.

The product from our low assay production area is produced batch-wise utilizing "limited safe" quantities for the particular assay with a maximum quantity of approximately 350 pounds for the lower assay material. Each 15 gallon drum, as it is filled in the final operation, contains no more than one "limited safe" batch.

The blending will be accomplished in a specially designed dust control hood. A maximum of 10 drums can be combined in a single operation in the following manner:

1. A single drum from a storage rack containing a "limited safe" batch will be placed in the hood. After closing the hood, the lid will be removed and the drum placed on a four wheel dolly in a canted position. The dolly rides on rails along the back of the hood.

2. In front of the rail carrier will be located ten stations, each separated from the next by a concrete slab 1 ft. thick, approximately 3 ft. high and 2 ft. wide to isolate each station from the next from a neutron interaction standpoint. An empty 15 gallon drum will be positioned in each station.

3. The dolly will be moved from station to station and an operator will transfer with a hand scoop, by volume, one-tenth of the product from the full drum into a drum in each of the receiving stations.

B-2

4. This operation will be repeated until the entire batch is blended or until the drums at each station are 65% filled. The individual drums having received the incremental parts of the blend will be fitted with a lid, removed from the hood, placed on a roller and tumbled to accomplish blending. At this point, the drums, if they contain a "limited safe" batch, will be accurately weighed and placed in birdcages and transferred to the storage vault awaiting shipment. In the cases where the 65% filled drum contains less than a "limited safe" batch, these drums will be transferred to a packaging station where the individual drums will be loaded, by weight. The "limited safe" quantity will not be exceeded in any drum. Following loading and weighing, the drum will be placed in a birdcage for transfer to the storage vault awaiting shipment.

The procedure for material movement control inside the blending building will be as follows:

1. Facilities

The blending room is approximately 20 x 50' and the entire area will be devoted to this single operation. The blender hood is on one wall. Steel storage racks will occupy two other walls. These racks are constructed to maintain 2 ft. edge to edge spacing between sides and top and bottom of the drums. One rack provides for two-high storage and one rack for three-high storage. Also in this area will be a dust controlled packaging hood which will be used in those cases where the batch size is greater than can be properly tumbled for adequate blending. Also in the same area will be two roller type blending machines for tumbling drums. These are so located that when two drums are being rotated at the same time, spacing of more than 2 ft. edge to edge is maintained.

2. Material Movement

Drums will be removed from the packaging station in the UO_2 production facility by fork truck and carried, one at a time, into the storage racks in the blender building. Brightly colored lines will be painted on floors as a guide to the fork truck operators so as to prevent the possible close approach of two "limited safe" batches. In the blender building, brightly colored lines will be painted on the floor in front of all storage racks and the blender hood as a guide to the operators to indicate the central floor area in which lateral movement of a drum will be permitted. This central area inside the colored lines will maintain at least a 2 ft. edge to edge space between the drum being moved and those in either the storage rack or the blender hood. All personnel will be carefully instructed as to the necessity of maintaining this spacing and the operating procedure will be rigorously enforced.

The filled drums from the blending hood loaded to approximately 65% by volume or less, depending upon assay, will be moved by hand dolly out of the individual stalls to the central area beyond the colored line and

moved to the rolling station. From the rolling station the drums will go into birdcages directly if the contents of the drum are a "limited safe" batch and to the packaging station if the drum contains less than a "limited safe" batch for filling to not more than the "limited safe" quantity. A scale is provided for accurate weighing checks.

We request your prompt consideration of this application and further request that if additional information is required we be informed by telephone or telegraph so that we may supply the needed additional information promptly.

Sincerely yours,

MALLINCKRODT CHEMICAL WORKS

W. M. Leaders

W. M. Leaders
Technical Director
Special Metals Division

WML:dj

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4 February 1958

Mr. Lyall Johnson
Licensing Division
U. S. Atomic Energy Commission
Washington, D.C.

SUBJECT: Special Nuclear Materials License No. SNM-33

Dear Mr. Johnson:

In the course of our business of preparing uranium compounds, it has become essential that we establish facilities for purification and recovery of our own internal waste residues. We are, therefore, proposing the construction of two facilities for solvent extraction to be used on internally generated scrap and recovery material such as floor sweepings, dust collector residues, etc., and unirradiated scrap product from external sources. One facility will be used for materials of assays from 5% upward to highly enriched. The other facility will be used for up to 5% enrichment. The facilities and the operating procedures to be utilized are described below:

1. 5% and Higher Enrichment

The process to be used will be as follows: (a) dissolve the scrap material in nitric acid, (b) extract the U values with TBP-kerosene solution (c) re-extract the U values from the TBP-kerosene solutions with water, (d) precipitate U values as ADU with ammonia, (e) filter and introduce the precipitate into our regular production line.

The equipment which will be used for high assay materials will be stainless steel beakers, and small tanks, batch operated, for dissolution in nitric acid. Two extraction devices will be utilized depending upon the assay and batch size. The first will be a small batch operated mixing tank where a single batch of U feed and the proper TBP-kerosene solution will be mixed. In all cases this equipment will have a maximum diameter of 5". The uranium solutions will contain no more than 300 g/l total U content.

The other equipment, which will be used for larger batches, at lower assays, will consist of counter-current flow columns for extraction and for reextraction. These columns will have a maximum diameter of 4". The material will be fed to the column either by gravity flow or with the use of small pumps. All equipment, columns, pumps, tanks, etc. necessary for the operation of this recovery unit will be housed in an appropriate fume hood. The

hood will be so designed that any spills will be retained in the hood up to a depth of 1". Appropriate slots will be placed in the hood to insure this maximum depth. Operating procedures will be rigidly enforced to insure that no more than one "limited safe" batch quantity of material will be in the extraction hood at any one time. These procedures will include pre-analysis of all scrap solutions and solids to determine their U content and weighing or measuring the proper quantity to make a "limited safe" batch.

2. For Enrichments up to 5%

For this area of the plant a large fume hood will be erected in which will be housed all of the extraction equipment. The equipment will consist of extraction columns of a maximum of 6" in diameter for the following operations: (a) extraction of U values into TBP-kerosene solution, (b) re-extraction from TBP-kerosene into water, and (c) continuous treatment of solvent. Also in this area will be feed preparation tanks, pumps, and product tanks of appropriate size. Following purification, the material will be precipitated from the final solution with ammonia or peroxide (H_2O_2) and filtered. The filter cake will then be introduced into the appropriate step of our regular production line. The extraction columns will be spaced to maintain approximately 2 ft. between edges to prevent interaction from a neutron standpoint. The tankage, however, is not amenable to such spacing; therefore, the operating procedure will call for not more than one "limited safe" batch of the particular assay material involved to be in the hood at any given time. A second batch of feed will not be made up without specific authorization of the supervisor in charge to insure against the possibility of double batching. The charge weight will be controlled by analysis of feed material prior to digestion and weighing the appropriate quantity into the digestion tank.

All solvents will be handled according to approved safety provisions and the lighting fixtures, motors, and other electrical equipment will be chosen in accordance with the standard practice for handling TBP-kerosene solutions. An exhaust fan on the hood discharging to the atmosphere will prevent the spread of solvent fumes to other areas of the plant. The floor of the hood is designed to be able to retain 1-1/2 tank volume of the largest vessel. The depth of the liquid in the floor will be limited to 2" by appropriate slot spill-overs for criticality control.

We are quite anxious to be able to offer scrap recovery service to the civilian nuclear industry and, therefore, urge prompt consideration of this application for modification of our License No. SNM-33 to include these two recovery areas. If we can be of further assistance in expediting this matter, do not hesitate to call upon us.

Sincerely yours,

MALLINCKRODT CHEMICAL WORKS

W. M. Leaders

W. M. Leaders
Technical Director
Special Metals Division

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4 February 1958

Mr. Lyall Johnson
Licensing Division
U. S. Atomic Energy Commission
Washington, D.C.

SUBJECT: Special Nuclear Materials License No. SNM-33

Dear Mr. Johnson:

This communication is our application for modification of our License SNM-33 to include a metal producing facility to handle enriched uranium of any assay.

The equipment to be used in this metal production facility was designed after careful considaration of the equipment that has been and is being used by AEC contract operators for the reduction of UF_4 into uranium metal enriched in the isotope U^{235} in excess of 90%. In every case where the uranium metal or intermediate materials are handled, we have attempted to copy as closely as possible the techniques currently used by these contractors. A detailed description of the various processing steps is given below:

1. Preparation of UF_4

Initial operation of our plant will utilize the preparation of UF_4 by hydrofluoriration of UO_2. The UO_2 will, in every case, be prepared in our approved oxide facility. A furnace has been designed which essentially duplicates those used in the Oak Ridge area for conversion of UO_2 to UF_4 with anhydrous HF. A special room, exterior to the main building, houses the HF supply and vaporiser. All pipes, fittings, and valves are of an approved design for safety using plastic enclosures around flanges and splash guards on all valve stems. A safety shower and eye fountain are located just outside the door as a further safeguard to protect the operators. All work in connecting and disconnecting HF cylinders and repair of piping will require that the operators and maintenance people wear approved plastic suits and air supply masks while performing these tasks. All operating personnel will be instructed in the precautions required for the handling of HF. Many of the personnel have had extensive experience from previous operations with this material. These experienced operators will be expected to assist in the training of the new operators.

The furnace in which the conversion of UO_2 to UF_4 will be made is housed in a totally enclosed hood so that any escaping HF vapors will be quickly removed from the building without contaminating occupied areas. The trays are designed so that their total cross sectional area is equivalent to a 5" cylinder. On this basis, we are using "safe" geometry in this retort for criticality control. The retort is so constructed that only two trays can be accommodated and our maximum furnace loading can be 2.650 kg of U content. The furnace cycle will be essentially as follows: After the UO_2 load has been introduced an inert atmosphere (nitrogen) will be turned on to check the gas-tightness of the flanged front and also to purge the air from the retort. The inert atmosphere will be maintained until the thermocouple in the retort is above 250°F. The HF supply will then be turned on and the heating continued for sufficient time to accomplish complete conversion to UF_4. During the cool-down cycle the inert atmosphere will again be used to sweep out the excess HF and protect the UF_4 from oxidation.

2. Loading the Reduction Reactor

Following the completion of the UF_4 conversion, the entire retort will be removed from the furnace and hood and physically connected to a glove box so that all handling of UF_4 will be done inside the dust control box. The trays will be removed from the retort, the UF_4 ground, weighed, and stored in a plastic container preparatory to loading the bomb. The quantities of calcium and iodine required for the reduction will be based on the UF_4 weight obtained from the furnace. These ingredients will then be mixed in the proper proportion with the UF_4 and the entire charge stored in a plastic container prior to loading in the reactor.

The reactor is of standard design utilized by AEC contractors. It is of heavy steel construction 5" I.D., 12-3/4" long with a flanged lid and an external thermocouple well. A prefired high density magnesium oxide thimble will be used as the reactor liner. The angular space between this thimble and the reactor shell will be packed with magnesium oxide sand. The blended charge will be introduced into the thimble and a copper gasket placed on the top of the bomb and the lid securely bolted in place. The bomb will then be evacuated and filled with argon to ambient pressure, sealed, and removed from the glove box through an airlock.

3. Firing

The ignition of the charge will be accomplished by heating in a high frequency induction coil. This coil is enclosed in a heavy metal hood, exhausted to the atmosphere as a precaution against room contamination from flange leaks or blowouts. The thermocouple on the side of the bomb will indicate firing conditions. Following the firing, the bomb will be removed from the furnace and placed in a table for cooling. This table, constructed of steel, has holes so that spacing between adjacent bombs will be 2 ft. edge to edge.

4. Unloading

After cooling, the entire bomb will be placed in a second glove box where the lid will be removed and the contents dumped and separated. Special openings are provided in the bottom of the glove box so that the slag liner and sand can be directly dumped into a drum, the metal button removed and mechanically cleaned. Following mechanical cleaning, the button will be pickled in acetic acid, washed with water and dried. A drill has been provided inside the glove box for sampling purposes.

5. Shipment

Buttons will be wrapped in polyethylene and shipped in a water-tight steel can rigidly fastened in the center of a birdcage. A maximum of 10 kg of U^{235} content in the metal will be shipped in any given birdcage.

These facilities have been adequately tested using normal isotopic assay uranium without encountering any difficulty from a mechanical, dust control, or safety standpoint. We believe that in as much as we have essentially copied designs which have been operated by Commission contractors for some years, we have a metal facility that is completely safe from a criticality standpoint as well as from the standpoint of safety and dust control.

We are, therefore, requesting approval of this facility for use with any assay of uranium in accordance with the operating procedures described herein. Since we are quite anxious to be able to offer service in the preparation of U metal to the civilian reactor industry, we are requesting as prompt consideration to this application as is possible. If additional information is required, we shall be quite happy to furnish it promptly.

Sincerely yours,

MALLINCKRODT CHEMICAL WORKS

W. M. Leaders

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Technical Director
Special Metals Division

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