## PATENT SPECIFICATION

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(72) Inventor WALTER PHILIP HOLLAND

# (54) IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF METAL OXIDES

(71) We, UNITED KINGDOM ATOMIC ENERGY AUTHORITY, London, a British Authority, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the production of metal oxides by thermal decomposition of their nitrates. The invention is considered to have particular application to the treatment of fissile material with which criticality problems can arise.

According to the present invention a method of producing a metal oxide from its nitrate by thermal decomposition comprises the steps of dispersing an aqueous solution of the nitrate on carbon fibre felt, heating to decompose the nitrate, and heating in the presence of free oxygen to oxidise the carbon felt.

Carbon felt prepared by the carbonisation of cotton wool or paper tissues has been found most satisfactory because of its low density and low impurity content.

In order to effectively disperse the nitrate solution on the carbon felt a wetting agent may be required. With nitrates of fissile 30 metals acetic acid and butyric acid have been found satisfactory as wetting agents. Other organic acids may also be used. Only a few drops are generally required on the carbon felt surface prior to the addition of the aqueous nitrate solution.

The method is generally suitable for the treatment of metal nitrates which decompose to oxide on heating. It has particular advantages for the production of plutonium and uranium oxides and may conveniently be used for the production of mixed plutonium/uranium oxides which find use as nuclear fuel. Oxides produced by the

method have a relatively high density.

[Price 25p]

The following are examples of ways of 45 carrying the invention into effect.

#### Example 1

Carbon felt between 4 mm and 10 mm thick and of density between 0.1 and 0.2 g/cc is disposed as a single layer in a tray or boat which may be of silica, platinum or alumina. The felt is wetted with acetic acid and aqueous plutonium nitrate solution of concentration 200 g/l is added until the felt is covered. The tray is then placed in a furnace at 600°C and the contents calcined first in a current of air and then in oxygen. With the oxygen current an exothermic reaction takes place and the temperature temporarily rises. The product is plutonium oxide PuO<sub>2</sub> containing less than 0.1% carbon.

#### Example 2

A carbonised cotton wool 1½" thick and 8" square is placed in an Inconel (Trade Mark) boat and 1 litre of uranyl nitrate solution is added. The boat is heated in an Inconel (Trade Mark) furnace firstly at 100°C in air enriched with 20% by volume of oxygen and then at 600°C in 100% oxygen. The product is a powder of U<sub>3</sub>O<sub>8</sub>.

#### Example 3

A carbon felt is saturated with a zinc nitrate solution containing 300 g/l of zinc. A silica boat containing 10 ml of this felt is heated in an alumina furnace in air at 700°C. The product is zinc oxide in a powdery form.

#### Example 4

A saturated solution of cobaltous nitrate 85 containing 300 g/l of cobalt is absorbed into 20 ml carbon felt in a silica boat. No additives were required. The felt was

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heated in an alumina furnace in air, the product was a brittle cake, resembling the carbon felt. When analysed, this cake contained 40 ppm carbon and 73.8% cobalt.

### WHAT WE CLAIM IS:-

- 1. A method of producing a metal oxide from its nitrate by thermal decomposition of the nitrate, the method comprising the steps of dispersing an aqueous solution of the nitrate on carbon fibre felt, heating to decompose the nitrate, and heating in the presence of free oxygen to oxidise the 15 carbon felt.
  - 2. A method according to claim 1 wherein the carbon fibre felt is the product of the carbonisation of cotton wool.
- 3. A method according to claim 1 20 wherein the carbon fibre felt is the product of the carbonisation of paper tissues.
  - 4. A method according to claim 1

wherein the nitrate is a nitrate of fissile metal.

- 5. A method according to claim 1 wherein dispersion of the nitrate on the carbon fibre felt is assisted by the addition of a wetting agent to the carbon felt.
- 6. A method according to claim 5 wherein the wetting agent is either acetic acid or butyric acid and the nitrate is a nitrate of a fissile metal.
- 7. A method of producing a metal oxide from its nitrate by thermal decomposition of the nitrate, substantially as hereinbefore 35 described in Example 1.
- 8. A method of producing a metal oxide from its nitrate by thermal decomposition of the nitrate, substantially as hereinbefore described in any of Examples 2—4.

J. Y. LE MASURIER, Chartered Patent Agent, Agent for the Applicants.

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