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PREPARATION OF HIGH-PURITY MAGNESIUM OXIDE

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Abstract

A procedure was developed for preparing high-purity magnesium oxide for use in growing single crystals needed in studies of radiation effects. The procedure consists in dissolving high-purity sublimed magnesium metal in nitric acid, extracting impurities from the solution into a TTA-Hexone solution, precipitating magnesium carbonate from the aqueous phase, and igniting the magnesium carbonate to magnesium oxide. Amounts of magnesium oxide from gram to pound quantities can be prepared.

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## INTRODUCTION

It was desired to obtain high-purity magnesium oxide for growing single crystals to be used in the study of radiation effects. Magnesium oxide was needed that contained less total transition elements than 20  $\mu\text{g/g}$  and no higher total concentration than 50  $\mu\text{g/g}$  of other elements that do not fit into the crystal lattice of magnesium oxide. Attempts to use Dowex 50W-X8 cation-exchange and Dowex 1-X10 anion-exchange resins for purifying the metal, after converting it to magnesium chloride, were both unsuccessful. A satisfactory procedure employing solvent extraction was developed.

## EXPERIMENTAL RESULTS AND DISCUSSION

The sublimed magnesium metal used as the starting material for preparing high-purity magnesium oxide contains aluminum, copper, iron, and zinc in concentrations that vary from 10 to 75  $\mu\text{g/g}$ . No other contaminants are present in significant concentrations. After these contaminants were removed from the metal by extraction into TTA-Hexone, high-purity magnesium oxide was prepared. The concentrations of the individual transition elements, as well as of other metal contaminants, in the oxide product were never more than 10  $\mu\text{g/g}$  and were usually less than 5  $\mu\text{g/g}$ .

Evaluation of Ion-exchange Procedures

Ion-exchange procedures seemed feasible for the purification of magnesium metal prior to producing the high-purity oxide. However, they proved unreliable.

Cation Exchange

Attempts to use Dowex 50W-X8 cation-exchange resin for purifying the metal, after the conversion of it to magnesium chloride, were unsuccessful because all cations present were adsorbed onto the resin column, and it was not possible to selectively elute magnesium from the column.

### Anion Exchange

An anion-exchange procedure was investigated in detail and showed some promise as a good way to purify the magnesium metal. A 3 to 4 M solution of magnesium chloride, adjusted to  $\text{pH } 5.0 \pm 0.5$ , was passed through a column of Dowex 1-X10 resin. Aluminum, copper, iron, and zinc were adsorbed on the resin. Pure magnesium carbonate was precipitated from the effluent by adjusting the pH to about 7.5 (not sufficiently alkaline to cause precipitation of magnesium hydroxide) with ammonium hydroxide and then adding a saturated solution of pure ammonium carbonate. This procedure was unsatisfactory because of the persistent occlusion of chloride, which is not eliminated by ignition. A sulfate or nitrate medium could not be used instead of the chloride medium because the metal-ion contaminants will not adsorb satisfactorily onto the resin from such media.

### Recommended Solvent-extraction Procedure

A satisfactory solvent-extraction procedure was developed for the preparation of high-purity magnesium oxide. The contaminants are extracted from a nitric acid solution of the magnesium metal into a TTA-Hexone solution. The pure magnesium is then precipitated from the aqueous phase as the basic carbonate, which is ignited to magnesium oxide.

### Reagents

1. Magnesium metal, high-purity, sublimed; available from Dow Chemical Co., Midland, Michigan.
2. Thenoyltrifluoroacetone (TTA); available from Columbia Organic Chemical Company, Inc., Columbia, S. C.
3. 4-Methyl-2-pentanone (Hexone); available from Distillation Products Industries, Rochester 3, N. Y., as reagent No. 416.

4. Ammonium hydroxide, "Baker Analyzed" grade; available from J. T. Baker Chemical Co., Phillipsburg, N. J.

5. Ammonium carbonate, "Baker Analyzed" grade; available from J. T. Baker Chemical Co., Phillipsburg, N. J.

#### Special Apparatus

Magnesia crucible (8 in. high x 4-1/2 in. OD x 3-7/8 in. ID); available from Lava Refractories Co., Pittsburg, Pa.

#### Detailed Procedure

Small pieces (3-to-5 g) of magnesium metal are dissolved, one at a time, in a Pyrex beaker that contains aqueous 6 to 8 M nitric acid to make about a liter batch of a 3 M solution of magnesium nitrate. This solution is adjusted to a pH of ~ 5.5 with either excess magnesium or ammonium hydroxide.

A 1-liter batch of 0.5 M TTA in Hexone is prepared by dissolving 111 g of thenoyltrifluoroacetone in Hexone.

An equal-volume single-pass extraction is made in a graduated cylinder (2-liter, Pyrex, stoppered). Use of this apparatus permits mixing and equilibration to be brought about gently by repeated inversions; thus the formation of an emulsion is avoided. After a 3-minute equilibration time, the two phases are poured into a clean 2-liter separatory funnel. The aqueous phase is drawn off into a 3-liter polyethylene beaker or bottle. The pH of the solution of pure magnesium is adjusted to about 7.5 (not alkaline enough to cause precipitation of magnesium hydroxide) with ammonium hydroxide. To the basic solution of magnesium nitrate is added a saturated solution of ammonium carbonate, in deionized water, in excess of the stoichiometric amount. After the mixture is digested for two hours on a steam bath, it is

cooled and filtered. The precipitate is transferred to a magnesia crucible and is ignited in a muffle furnace for four hours at 900 to 1000°C. The pure MgO is removed from the furnace, cooled, and placed in a clean polyethylene bottle for storage.

This process can be scaled proportionally to produce pound quantities of high-purity magnesium oxide. In addition, the TTA-Hexone phase that remains after one extraction may be reused to purify two or three more solutions of sublimed magnesium metal since its chelating capacity is not exceeded after one use.

#### Purity of the MgO Product

The average concentration of contaminants found in two batches (~1000 g total) of MgO product are listed in Table I.

Table I. Contaminants Remaining in High-purity MgO

<u>Element</u>	<u>Concentration, <math>\mu\text{g}</math> per gram MgO</u>
Al	< 4
Co	< 3
Cr	< 4
Cu	< 4
Fe	< 4
Mn	None
Mo	< 5
Ni	< 3
Pb	< 10
Si	< 5
Sn	< 5
Ti	< 2
Zn	< 7
Li	< 1
Na	< 10
K	< 5

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Distribution

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