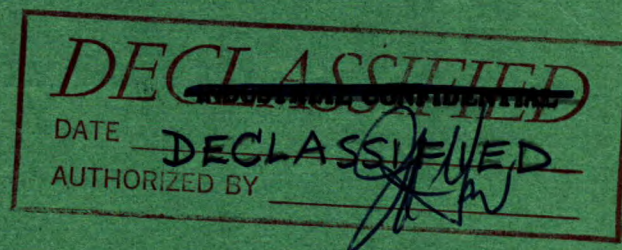


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DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 67-23

**FERRITES: PART II. INVESTIGATION OF THE  
CO-PRECIPIATION METHOD OF  
PREPARATION OF PERMANENT-MAGNET  
TYPE FERRITE POWDERS**

by

SUTARNO AND W. S. BOWMAN

MINERAL SCIENCES DIVISION

COPY NO. 0



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FERRITES: PART II. INVESTIGATION OF THE CO-PRECIPITATION  
METHOD OF PREPARATION OF PERMANENT-MAGNET TYPE  
FERRITE POWDERS

by

Sutarno\* and W.S. Bowman\*\*

SUMMARY OF RESULTS

Permanent-magnet type ferrite powders with the composition  $(\text{Ba, Sr, Pb})\text{O} \cdot n\text{Fe}_2\text{O}_3$ , with  $n$  near to 6, were prepared by the co-precipitation method. Barium, strontium and lead were precipitated as carbonates and iron as the hydroxide. It was found that the formation of both barium and strontium ferrites had started at  $750^\circ\text{C}$  and was complete at  $1050^\circ\text{C}$ . The formation of lead ferrite started at  $700^\circ\text{C}$  and was complete at  $900^\circ\text{C}$ . No impurities were introduced during the co-precipitation process, but some segregation of the constituents was observed during settling. DTA, TGA, chemical and spectrographic analyses were used to evaluate the properties of the co-precipitated powders; X-ray diffraction analyses of the products were also conducted to follow the progress of the reactions.

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## INTRODUCTION

The effect of chemical stoichiometry and of purity both on the ceramic and the magnetic properties of permanent-magnet type ferrites has been reported by a number of previous investigators. A summary of their published findings has been given in Part I of this series of reports (1)\*.

The classical method of preparation of permanent-magnet type ferrite powders is by mixing the components as powders in a ball mill and reacting them by means of heating. With this method of mixing, it is difficult to control the reproducibility of the stoichiometry and purity of the product. Furthermore, the calcined powder produced by this method usually consists of particles larger than the single-domain size required to make good magnetically oriented specimens. A second milling is, therefore, necessary. This, of course, increases the discrepancies in the chemical composition.

In an attempt to overcome these difficulties, the possibility of using a chemical co-precipitation method for preparation of such powders is being investigated (2). The first method studied was the co-precipitation of the barium, strontium and/or lead as carbonates with the iron as hydrated iron oxide.

The merits of the co-precipitated powders were determined by chemical, spectrographic, X-ray diffraction (XRD), differential thermal (DTA) and thermogravimetric (TGA) analyses.

## EXPERIMENTAL

### 1. Full Co-Precipitation Method

Metal nitrates were found to be the most suitable raw materials for this purpose because they are readily soluble in water and the solutions are stable over a long period of time. Reagent grade chemicals were used without purification. The manufacturers' supplied analyses are listed in Appendix I (see page 12). The nitrate solutions were analysed separately before mixing, using standard gravimetric techniques (see Appendix II). The analysed solutions were then mixed volumetrically to give the desired  $Ba^{++}:Sr^{++}:Pb^{++}:Fe^{+++}$  molar ratios. The hydrated oxide of iron and the carbonates of barium and strontium and the basic carbonate of lead,

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\* For references, see page 11.

were co-precipitated from the blended metal nitrate solution. For small-scale work,  $\text{NH}_4\text{OH}$  and  $\text{NH}_4\text{HCO}_3$  were used as precipitants. For large-scale precipitations (1 lb of product or more),  $\text{NH}_3$  diluted with  $\text{N}_2$  and  $\text{CO}_2$  were used in the pilot-plant equipment in the Extraction Metallurgy Division. A schematic diagram of this equipment is shown in Figure 1.

Large-scale co-precipitation was accomplished by spraying the mixed nitrate feed solution into a plastic tank containing distilled water into which was bubbled dilute  $\text{NH}_3$  and  $\text{CO}_2$  through two separate gas diffusers. The pH was maintained at a value above 7 by controlling the flow of  $\text{NH}_3$ . The temperature was kept constant at  $65^\circ\text{C}$  by enclosing the precipitation tank in a steam jacket and controlling the flow of steam. The steam jacket has since been replaced by two 1000-watt immersion heaters which are much easier to control than the steam flow. The precipitation was carried out at  $65^\circ\text{C}$  in order to flocculate the fine hydrated ferric oxide. This procedure makes subsequent decantation and filtering easier, and also ensures complete precipitation of the iron. The disadvantage of co-precipitation at this elevated temperature is that the carbonates, which are crystalline, grow to a greater extent than they do at room temperature.

In order to maintain a constant level in the precipitation tank, the slurry was syphoned continuously into a settling tank. The precipitate was washed three times by decantation, filtered through a Buchner funnel, washed once with methanol and then dried overnight at  $110^\circ\text{C}$  on a pyrex tray.

## 2. Drying Techniques

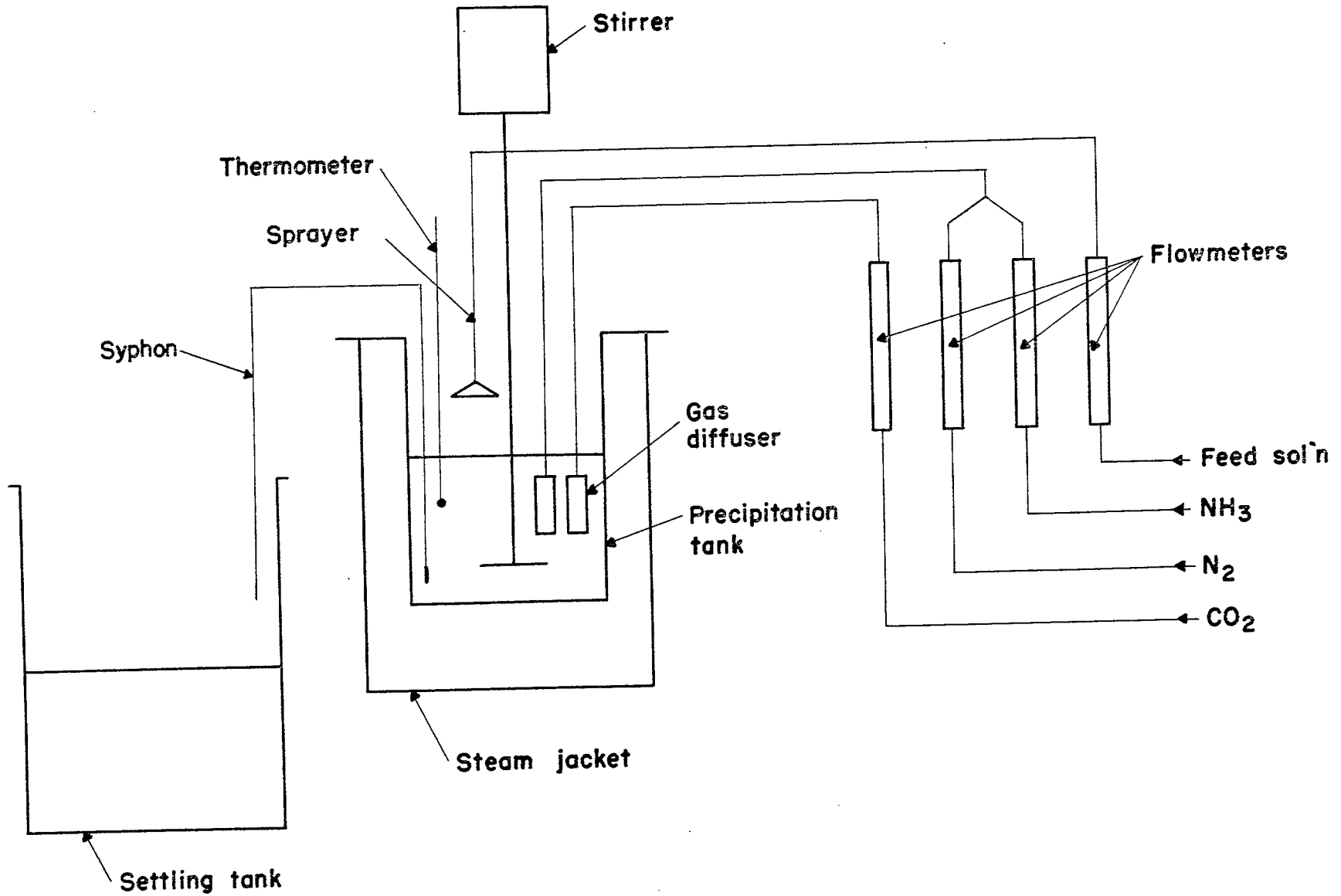
The barium (or other) carbonate and hydrated iron oxide mixture produced by the full co-precipitation method had the semi-colloidal properties of an iron hydroxide slurry, which made it difficult to filter. The mass shrank during drying and formed a hard lump that required to be ground before calcination. To overcome this difficulty, various drying techniques have been tried.

### a) Spray drying

The slurry was washed by decantation and dried in a spray-dryer with the sprayer rotating at 38,000 rpm with simultaneous electrical heating of the spray to about  $250^\circ\text{F}$ . The dried powder was collected by means of a cyclone dust collector.

### b) Freeze drying

This experiment was carried out on a laboratory scale. About 100 ml of slurry containing about 5% solids was poured into a 600 ml pyrex bulb. The bulb was equipped with a cover that



**FIGURE 1 SCHEMATIC DIAGRAM OF PRECIPITATION EQUIPMENT**

could be connected to a vacuum chamber. The vacuum was created by a vacuum pump with a liquid nitrogen trap. The slurry in the bulb was frozen by immersing the bulb in the liquid nitrogen. The bulb was rotated while the slurry was freezing, so that the mass formed a thin layer of ice on the inside wall of the bulb. When the slurry was completely frozen, the bulb was immersed in the liquid nitrogen for some time to create cracks on the ice layer, thus increasing its exposed surface area. The bulb was then continuously evacuated until complete dryness was attained. A heat lamp was used to increase the evaporation rate, but it was so operated that it would not melt the ice.

### c) Introduction of some organic reagents

The particles in the slurry were coated with organic compounds following the procedure developed by Puddington and Sirianni (3). Approximately 150 ml of the slurry (containing about 5% solids) was mixed with about 10 drops of oleic acid and 2 drops of oil. The mixture was stirred vigorously for about 15 minutes until the solid material formed small balls and separated completely from the water. The water was then removed either by filtration or by decantation and the solid balls were ignited at about 500°C in an oxygen-rich flame.

Other surface-active organic compounds, such as octadecylamine acetate, mixed with kerosene in various quantities were also tried as a replacement for oleic acid.

### 3. DTA, TGA and XRD Examinations

DTA, TGA and X-ray diffraction investigations of some of the co-precipitated powders were carried out in an attempt to determine the completion of the reaction, and thus the calcination temperature to be used.

The DTA examinations were conducted in air at a heating rate of 12 deg C per minute, up to 1100°C.  $\alpha$ -Alumina was used as the standard reference material. The sample was held in a palladium metal sample holder and Pt. vs Pt:13% Rh thermocouples were used for both sample and differential temperature measurements. The differential EMF and the sample temperature were simultaneously recorded on a two-pen Leeds and Northrup recorder, the former after amplification and the latter directly.

The TGA examinations were carried out using a Stanton Automatic Recording Thermobalance in an air atmosphere. The sample weighing about 0.8 g, contained in small alumina crucible, was heated to about 1200°C with a heating rate of 385 deg C per hour.

X-ray diffraction patterns of the samples at various stages of heating were taken to identify the phases present. The photographs were taken with a 114.7 mm diameter Debye-Scherrer camera using filtered Co K radiation.

## RESULTS AND DISCUSSION

The co-precipitated powders consist of iron as ferric hydroxide and barium, strontium and/or lead as carbonates. Optical microscopic examination showed crystalline carbonate particles of about 5 $\mu$  or less diameter surrounded by the amorphous iron hydroxide. The co-precipitations were quantitative. Tests for iron (using ammonium thiocyanate) and for barium, strontium and/or lead (using sulphate, carbonate or chromate, respectively) in the filtrates showed negative results. The small-size co-precipitation is therefore satisfactory. In larger batches, however, the powder exhibited segregation. This is shown by the results of chemical analyses listed in Table 1. With the first five samples, the powders were ground and passed through a 100 mesh screen before being submitted for analysis. The analysed ratios of Fe<sub>2</sub>O<sub>3</sub>/MO were within  $\pm$  1% of their intended compositions, which is probably within the limits of accuracy of the analyses. The grinding of the sample does, of course, create the possibility of introducing impurities from the grinder material and this would defeat one of the purposes of the co-precipitation techniques. The other samples were submitted to analysis without being previously ground. The results showed discrepancies of as high as 8% between analysed and theoretical Fe<sub>2</sub>O<sub>3</sub>/MO values. These discrepancies are apparently a result of segregation occurring during the settling, due to the difference in size of the carbonates and hydroxide particles.

The spectrographic analyses (Table 2) showed that the purity of the powders was comparable with that of the reagent grade chemicals of this type. No appreciable additional impurities were introduced during the co-precipitation process.



TABLE 1

## Chemical Analyses of Co-Precipitated Ferrite Powders

| Sample No. | Intended Composition                                      | Analysed mole ratio normalized to intended F figure |      |      |      | F/M   |        |    |
|------------|---|---|------|------|------|-------|--------|----|
|            |   | F   | B    | S    | P    | Anal. | Theor. | Δ% |
| 1.2        | P. 5.9 F  | 5.9   | -    | -    | 0.99 | 5.9   | 5.9    | +1 |
| 6.12       | B. 5.9 F  | 5.9   | 0.99 | -    | -    | 6.0   | 5.9    | +1 |
| 21.2       | S. 5.9 F  | 5.9   | -    | 0.99 | -    | 6.0   | 5.9    | +1 |
| 4.1        | B <sub>0.6</sub> P <sub>0.4</sub> .5.9 F                  | 5.9   | 0.60 | -    | 0.41 | 5.8   | 5.9    | -1 |
| 13.1       | B <sub>0.2</sub> S <sub>0.4</sub> P <sub>0.4</sub> .5.9 F | 5.9   | 0.20 | 0.41 | 0.40 | 5.8   | 5.9    | -1 |
| 6.20       | B.6.0 F   | 6.0   | 1.01 | -    | -    | 5.9   | 6.0    | -1 |
| 1.3        | P.6.0 F   | 6.0   | -    | -    | 1.10 | 5.5   | 6.0    | -8 |
| 1.4        | P.5.8 F   | 5.8   | -    | -    | 1.01 | 5.8   | 5.8    | -1 |
| 4.2        | B <sub>0.6</sub> P <sub>0.4</sub> .6.0 F                  | 6.0   | 0.55 | -    | 0.41 | 6.2   | 6.0    | +4 |
| 6.13       | B.5.8 F   | 5.8   | 0.97 | -    | -    | 6.0   | 5.8    | +3 |
| 6.14       | B.6.0 F   | 6.0   | 1.03 | -    | -    | 5.8   | 6.0    | -3 |
| 15.1       | B <sub>0.6</sub> S <sub>0.4</sub> .5.9 F                  | 5.9   | 0.58 | 0.40 | -    | 6.0   | 5.9    | +2 |
| 15.2       | B <sub>0.6</sub> S <sub>0.4</sub> .6.0 F                  | 6.0   | 0.54 | 0.40 | -    | 6.4   | 6.0    | +6 |
| 16.1       | S <sub>0.6</sub> P <sub>0.4</sub> .5.9 F                  | 5.9   | -    | 0.58 | 0.40 | 6.0   | 5.9    | +2 |
| 16.2       | S <sub>0.6</sub> P <sub>0.4</sub> .6.0 F                  | 6.0   | -    | 0.57 | 0.40 | 6.2   | 6.0    | +3 |
| 21.3       | S.6.0 F   | 6.0   | -    | 0.94 | -    | 6.4   | 6.0    | +6 |
| 21.4       | S.5.8 F   | 5.8   | -    | 0.97 | -    | 6.0   | 5.8    | +3 |

NOTE:- The following abbreviations are used in this table:

P = PbO; B = BaO; S = SrO; F = Fe<sub>2</sub>O<sub>3</sub>; M = (Ba + Sr + Pb)O.  
 Δ = deviation.

TABLE 2

## Semi-quantitative Spectrographic Analyses of Co-precipitated Powders

| Sample No. | Composition   | Si %  | Mg %  | Pb %  | Cr %  | Fe % | Al %  | V %   | Ca % | Mo % | Cu %  | Na % | Ti %  | Sr % | Ba %  |
|------------|---|-------|-------|-------|-------|------|-------|-------|------|------|-------|------|-------|------|-------|
| 1.2        | P.5.9 F   | 0.060 | 0.007 | PC    | 0.05  | PC   | 0.004 | 0.009 | 0.01 | 0.02 | 0.020 | 0.10 | 0.001 | ND   | ND    |
| 4.1        | B <sub>0.6</sub> P <sub>0.4</sub> .5.9 F                  | 0.040 | 0.003 | PC    | 0.04  | PC   | 0.003 | 0.002 | 0.03 | 0.01 | 0.020 | 0.09 | 0.001 | ND   | PC    |
| 6.12       | B.5.9 F   | 0.050 | 0.008 | 0.060 | 0.05  | PC   | 0.003 | 0.003 | 0.01 | 0.02 | 0.020 | ND   | 0.001 | ND   | PC    |
| 21.2       | S.5.9 F   | 0.050 | 0.007 | 0.008 | 0.05  | PC   | 0.003 | 0.003 | 0.01 | 0.02 | 0.020 | ND   | 0.001 | PC   | ND    |
| 13.1       | B <sub>0.2</sub> S <sub>0.4</sub> P <sub>0.4</sub> .5.9 F | 0.040 | 0.009 | PC    | 0.05  | PC   | 0.003 | 0.002 | 0.01 | 0.02 | 0.020 | ND   | 0.001 | PC   | PC    |
| 15.2       | B <sub>0.6</sub> S <sub>0.4</sub> .6.0 F                  | 0.010 | 0.001 | 0.020 | 0.01  | PC   | 0.003 | -     | -    | -    | 0.005 | -    | 0.001 | PC   | PC    |
| 15.1       | B <sub>0.6</sub> S <sub>0.4</sub> .5.9 F                  | 0.007 | 0.005 | ND    | 0.01  | PC   | ND    | -     | -    | -    | 0.003 | -    | ND    | PC   | PC    |
| 1.3        | P.6.0 F   | 0.010 | 0.005 | PC    | 0.01  | PC   | ND    | -     | -    | -    | 0.005 | -    | 0.001 | ND   | ND    |
| 1.4        | P.5.8 F   | 0.009 | 0.005 | PC    | 0.01  | PC   | ND    | -     | -    | -    | 0.004 | -    | 0.001 | ND   | ND    |
| 6.13       | B.5.8 F   | 0.010 | 0.005 | 0.040 | 0.01  | PC   | ND    | -     | -    | -    | 0.003 | -    | ND    | ND   | PC    |
| 6.14       | B.6.0 F   | 0.008 | 0.020 | 0.010 | 0.01  | PC   | ND    | -     | -    | -    | 0.005 | -    | ND    | ND   | PC    |
| 16.1       | S <sub>0.6</sub> P <sub>0.4</sub> .5.9 F                  | 0.008 | 0.010 | PC    | 0.01  | PC   | 0.003 | -     | -    | -    | 0.004 | -    | 0.001 | PC   | <0.05 |
| 16.2       | S <sub>0.6</sub> P <sub>0.4</sub> .6.0 F                  | 0.010 | 0.004 | PC    | 0.001 | PC   | ND    | -     | -    | -    | 0.006 | -    | ND    | PC   | <0.05 |
| 21.3       | S.6.0 F   | 0.010 | 0.005 | 0.020 | 0.01  | PC   | ND    | -     | -    | -    | 0.004 | -    | ND    | PC   | <0.05 |
| 21.4       | S.5.8 F   | 0.008 | 0.010 | 0.010 | 0.01  | PC   | ND    | -     | -    | -    | 0.004 | -    | ND    | PC   | <0.05 |

ND = None detected. PC = Principal constituent.

Of the various methods of drying, only the freeze-drying method did not promote segregation. The pre-calcined powder dried by this method has a surface area of about  $5 \text{ m}^2/\text{g}$ . X-ray diffraction examination of this powder, calcined at  $1100^\circ\text{C}$  without mechanical mixing, showed only very faint or no foreign lines. However, at present, the freeze-drier equipment available has a capacity of only a few grams per day. This makes the drying operation by such a technique impractical for a large batch.

The DTA patterns of the co-precipitated powders are more complicated than those of dry-mixed powders. X-ray diffraction showed that the formation of the hexagonal ferrites of both barium and strontium had started at about  $750^\circ\text{C}$ . The bulk of the ferrite formation, however, was not completed until  $1050^\circ\text{C}$ , which is about the same temperature as is obtained for dry-mixed samples. The TGA showed that the weight loss was not as sharp as with dry-mixed powders. A sample calcined at  $1100^\circ\text{C}$  for 20 minutes had sintered to a greater extent than a dry-mixed sample treated similarly. The iron oxide in the co-precipitated powder is, apparently, very reactive, but the carbonates, being much larger in size, cannot match this reactivity. All foreign lines on the X-ray patterns could be identified as being due either to witherite ( $\text{BaCO}_3$ ), strontianite ( $\text{SrCO}_3$ ) or hematite ( $\text{Fe}_2\text{O}_3$ ), except for one with a d-value of  $3.14 \text{ \AA}$ , which presumably belongs to  $\text{BaO}\cdot\text{Fe}_2\text{O}_3^*$ .

Another foreign line, with a d-value of  $3.82 \text{ \AA}$  often appeared both on the barium and strontium ferrite patterns. This foreign line, which has not yet been identified, disappeared after repeated calcination. Both the  $\{002\}$  and  $\{004\}$  lines were sometimes visible at a reasonable strength, especially for lead ferrite; this was probably caused by an accidental orientation. Neither SrO nor BaO lines were observed.  $\text{BaO}\cdot 2\text{Fe}_2\text{O}_3$ , which has been reported to exist as an intermediate product (characterized by a line having  $d = 3.40 \text{ \AA}$ ) (4) was not observed.

All these foreign lines, except those due to hematite disappeared as the calcination temperature reached  $1000^\circ\text{C}$ . The hematite lines persisted until the sample was either calcined to about  $1200^\circ\text{C}$  or higher, or was given repeated calcinations at  $1100^\circ\text{C}$  with mechanical mixing in between.

This wide range of reaction temperature may be caused by the wide range in particle size of the carbonates, as observed under the microscope, combined with very reactive freshly co-precipitated iron oxide. In other words, the size of the carbonate particles seems to be the controlling factor of the rate of reaction.

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\*  $\text{BaO}\cdot\text{Fe}_2\text{O}_3$  was prepared by dry mixing and calcining at  $1300^\circ\text{C}$  for periods of one hour each. The strongest line of its X-ray pattern has the d-value of  $3.14 \text{ \AA}$ .

The lead ferrite exhibits a different behaviour from its barium and strontium counterparts. Lead carbonate and basic carbonate are decomposed at 315°C and 400°C, respectively (5). The DTA curves for these compounds showed no peak beyond 600°C. The TGA curve showed that the weight loss was complete at about 580°C and that no further loss occurred until 1100°C was reached.

Co-precipitated lead ferrite powder was calcined at different temperatures for 1 hour and X-ray powder photographs of the products were taken. The powder calcined at 600°C showed only hematite lines. (The same procedure was repeated using powder prepared by the slurry method, and very weak lead oxide (PbO) lines were detected in addition to the hematite lines.) At 700°C the ferrite lines started to appear; at 800°C the ferrite lines became stronger than the hematite lines and by 900°C, only ferrite lines were visible. The X-ray pattern of the ferrite did not change with the rising calcination temperature from 900 to 1170°C. However, the pattern of the powder calcined at 1200°C for 12 hours showed very strong hematite lines and only weak ferrite lines. This indicated that appreciable lead loss had occurred at this temperature.

## CONCLUSIONS

From the foregoing discussion of the results of the preparation of barium, strontium and lead hexaferrite powders by the co-precipitation method as described on pages 5 to 9, the following conclusions can be drawn:

- 1) The co-precipitation is quantitative. Therefore, for a small-scale sample preparation, this procedure produces a powder with the desired chemical composition and purity.
- 2) In a large-scale preparation, however, some segregation does occur. This segregation is probably caused by the differences between the particle sizes of barium, strontium and lead carbonates and that of the hydrated iron oxide.
- 3) The formation of barium and strontium ferrite may be started at a slightly lower temperature than with a sample prepared by dry mixing, but the completion of the reaction occurs at about the same temperature (about 1050°C). At this temperature, the particles start to sinter. Thus, some means of grinding is necessary if single-domain particles are desired for use in the later stages of processing to produce an oriented magnetic ferrite body.

- 4) A small amount of hematite (estimated at about 1-3%) was still present in the samples calcined at 1150°C for both barium and strontium ferrites. This may be caused by the lack of intimate mixing due to the differences in particle sizes of the reacting materials. This small quantity of hematite will disappear only after repeated calcinations at 1100°C or after calcination at about 1250°C.
- 5) The lead ferrite was formed at a much lower temperature than was its barium counterpart. The reaction was complete at 900°C. However, at 1200°C in an air atmosphere, the sample started to lose some of its lead. No intermediate product was detected in this reaction.
- 6) The differences between the reaction temperature for lead ferrite and those for its barium and strontium counterparts will presumably create some difficulties in the formation of the mixed Ba-Sr-Pb ferrite solid solutions.
- 7) Because of its high purity and reactivity, the samples prepared by the co-precipitation method may have some superior properties for certain purposes such as growing single crystals, etc.

#### PROPOSED FURTHER INVESTIGATION

In view of the above conclusions, the following investigations, in continuation of the foregoing experiments, are being carried out:

1. A "semi-co-precipitation" method for the preparation of barium, strontium and lead ferrite powders is being studied. In this method, the carbonates of the barium, strontium and lead are precipitated on a suspension of the iron oxide powder particles.
2. A milling method that will not introduce impurities into the calcined powder is being investigated.
3. The effects of the particle size of barium and strontium carbonates on the formation of ferrite are being studied.

A subsequent report will deal with the results of these studies when they are completed.

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APPENDIX I

Raw Materials:

The raw materials employed in these experiments were reagent-grade chemicals used without further purification. Ordinary laboratory distilled water was used and the contacts between the samples and metals were minimized by using plastic equipment wherever possible. The following are the batch analyses of the raw materials, as supplied by the manufacturers.

1. Barium Nitrate. (Fisher Scientific Company)

|  |         |   |
|--|---------|---|
| Chloride (Cl)  | 0.001   | % |
| Iron (Fe)  | 0.00004 | % |
| Insoluble Matter                                       | 0.005   | % |
| Substance not ppt'd. by H <sub>2</sub> SO <sub>4</sub> | 0.030   | % |
| Heavy Metals (as Pb)                                   | 0.0001  | % |
| Strontium (Sr)   | 0.05    | % |
| pH of 5% Solution at 25°C                              | 5.2     |   |

2. Strontium Nitrate. (Fisher Scientific Company)

|  |           |      |
|--|-----------|------|
| Assay Sr(NO <sub>3</sub> ) <sub>2</sub>      | min. 99.0 | %    |
| Maximum Limits of Impurities:                |           |      |
| Insoluble                                    | 0.010     | %    |
| Loss on Drying at 105°C                      | 0.10      | %    |
| Chloride (Cl)                                | 0.002     | %    |
| Sulphate (SO <sub>4</sub> )                  | 0.005     | %    |
| Calcium (Ca)                                 | 0.002     | %    |
| Barium (Ba)                                  | To pass   | Test |
| Magnesium and Alkali Salts<br>(as Sulphates) | 0.15      | %    |
| Heavy Metals (as Pb)                         | 0.0005    | %    |
| Iron (Fe)                                    | 0.0005    | %    |
| pH of 5% Solution at 25°C                    | 5.0-7.0   |      |

3. Lead Nitrate. (Fisher Scientific Company)

|  |        |   |
|--|--------|---|
| Insoluble Matter   | 0.002  | % |
| Chloride (Cl)  | 0.0002 | % |
| Copper (Cu)  | 0.001  | % |
| Iron (Fe)  | 0.0008 | % |
| Substances not ppt'd. by<br>H <sub>2</sub> S (as SO <sub>4</sub> ) | 0.03   | % |

4. Ferric Nitrate. (Fisher Scientific Company)

|   |        |   |
|---|--------|---|
| Insoluble Matter                            | 0.002  | % |
| Chloride (Cl)                               | 0.0002 | % |
| Sulphate (SO <sub>4</sub> )                 | 0.005  | % |
| Substances not ppt'd. by NH <sub>4</sub> OH | 0.002  | % |



## APPENDIX II

### Chemical Analyses of Raw Material Solutions

The aqueous solutions of the raw materials were assayed separately before mixing by well-established gravimetric methods (6). The co-precipitated powders were submitted to the Analytical Chemistry Sub-Division, Mineral Sciences Division, for chemical analyses for their major constituents, and for the detection of any impurities present, by semi-quantitative spectrographic analysis. The detailed procedures employed in assaying the raw materials were as follows:

#### 1. Analysis of Barium Nitrate Solution

10 ml of barium nitrate solution, containing about 0.3 mole of barium nitrate per litre, was transferred to a 600 ml beaker and diluted to about 300 ml. 5 g of sulphamic acid crystals were then added. The mixture was digested for about 1 hour on a hot plate, filtered through a pre-weighed Gooch crucible (No: 3001), and then dried and ignited at 900°C and the precipitate weighed as BaSO<sub>4</sub>.

#### 2. Analysis of Strontium Nitrate Solution

10 ml of strontium nitrate solution, containing about 1.1 mole of strontium nitrate per litre, was diluted to 100 ml. 10 ml of a 0.5 mole/l solution of KH<sub>2</sub>PO<sub>4</sub> was added. The mixture was heated to the boiling point and a 1 N solution of KOH was added dropwise until pH ≈ 7 was reached. The colloidal slurry was boiled continuously until it became a crystalline precipitate. The slurry was then filtered through a pre-weighed Gooch crucible (No. 3001), dried at 120°C and the precipitate weighed as SrHPO<sub>4</sub>.

#### 3. Analysis of Lead Nitrate Solution

25 ml of lead nitrate solution, containing about 0.8 mole of lead nitrate per litre, was diluted to about 200 ml. 1:4 acetic acid was then added until the solution was distinctly acid. The solution was then heated to the boiling point. 25 ml of 4% K<sub>2</sub>CrO<sub>4</sub> solution was added and the heating was continued until the precipitate settled. The precipitate was then filtered, washed with a 1% ammonium acetate solution, dried at 120°C, and weighed as PbCrO<sub>4</sub>.

4. Analysis of Iron Nitrate Solution

25 ml of iron nitrate solution, containing about 0.3 mole of ferric nitrate per litre, was diluted to about 300 ml. Ammonium hydroxide was added and the slurry was heated until the precipitate settled and the supernatant liquid was absolutely clear. The slurry was then filtered on an ashless filter paper, ignited at 900°C and weighed as  $\text{Fe}_2\text{O}_3$ .

APPENDIX III

ELECTRONIC CERAMICS

EGRDC RESEARCH PROJECT C 73

Identification of Mines Branch Personnel

Advisory Committee

Mr. Ian F. Wright, MPD\*, Chairman  
Mr. W. A. Gow EMD  
Dr. N. F. H. Bright, MSD  
Mr. J. G. Brady, MPD  
Mr. V. A. McCourt, MPD

Operational

Function

|                         |  |
|-------------------------|--|
| Mr. V.M. McNamara, EMD  | Pilot plant ceramic powder preparation           |
| Mr. J.C. Ingles, EMD    | Control analyses                                 |
| Dr. A.H. Webster, MSD   | Sintering and structural studies                 |
| Mr. V.A. McCourt, MPD   | Lapidary and electroding                         |
| Mr. Ian F. Wright, MPD  | Ceramic engineering                              |
| Mr. T.B. Weston, MPD    | Electronic test methods and component evaluation |
| Mr. W.R. Inman, MSD     | Wet chemical analytical methods and analyses     |
| Dr. A.H. Gillieson, MSD | Spectrographic analyses                          |
| Dr. E.H. Nickel, MSD    | Sample preparation for petrographic studies      |

\*MPD - Mineral Processing Division  
EMD - Extraction Metallurgy Division  
MSD - Mineral Sciences Division

Ian F. Wright  
Project Co-ordinator

John Convey  
Director, Mines Branch