DETERMINATION OF IMPURITIES IN BERYLLIUM OXIDE USED FOR BERYLLIUM METAL PRODUCTION (1)

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1. Introduction

We have studied on the beryllium metal production by calcium metal reduction (in alloying process) of beryllium oxide. Impurities in beryllium oxide and metallic calcium as reductant enter mostly into metallic beryllium produced by reduction almost, so we must check the impurities in beryllium oxide and metallic calcium precisely.

We tried the spark spectroscopic analytical method for beryllium oxide sample and calcium oxide sample to determine impurities in them.

The impurities determined in the 1st report are as follows;

Ferric oxide, magnesium oxide, calcium oxide, silicon dioxide in beryllium oxide and ferric oxide and magnesium oxide in calcium oxide, where calcium oxide is produced by burning of metallic calcium.

2. Experimental Apparatus

The spectrometer used is a middle qualz spectrometer QF-60 type with a spark producing apparatus (the condense-spark spectrometric analytical method). The principal mechanics is shown in Fig. 1.

A microphotometer was used in the case of quantative analysis. The principal mechanics is shown in Fig. 2.

3. Experimental Procedure

In taking the anaiytical spectrographs, the conditions used were as follow;

Potential of electric source	100 volt
Transformer	1st: 100 volt, 2nd: 15000 volt
Potential	1st: 30 volt, 2nd: 4500 volt
Interval between electrodes	0.8 mm
Exposing time	2–15 min

We used a graphite pole as a electrode, which was bouled to be 2 mmϕ and 2 mm depth and burried with powder sample and charged in the spark producing apparatus.

In qualitative analysis, we used a spectroscopic photograph 20 times enlarger with a spectrograph dry plate for qualitative determination, for the following lines which are the strongest lines regarding each permanent line of elements;
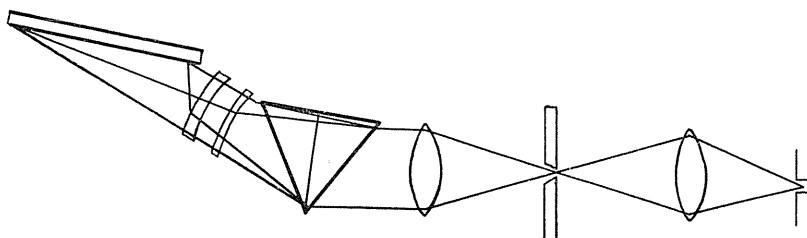
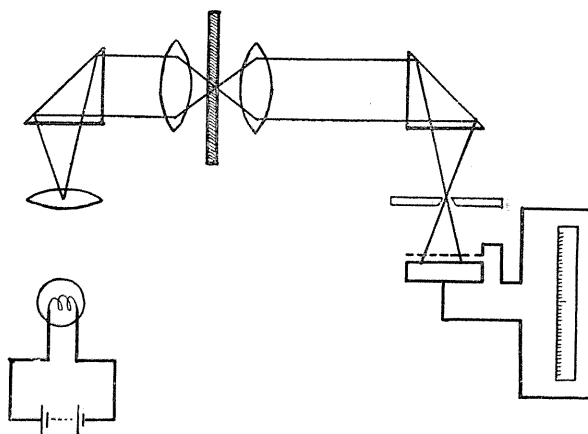FIG. 1. Principal system of spectrometer.

FIG. 2. Principal system of microphotometer.

Beryllium	:	3130.4 Å
Calcium	:	3930.7 Å
Silicon	:	2881.6 Å
Magnesium	:	2795.5 Å
Iron	:	2599.4 Å

   In quantitative analysis, for quantitative determination of impurities in samples we made a relative spector line intensity-quantity (ppm in sample) curve, or calibration curve, by observing the spector intensity to the specified impurity quantity in ppm. Experimental samples were prepared by pure beryllium oxide or calcium oxide or calcium carbonate plus pure impurity compound weighed very precisely.

   We used favourable spector lines among the permanent spector lines for quantative determination as follows:

Calcium	:	3968.5 Å
Silicon	:	2381.6 Å
Magnesium	:	2852.1 Å
Iron	:	2599.4 Å

Using the intensity ratio between the spector intensity and the baseline intensity, we selected the following bright lines as base lines;
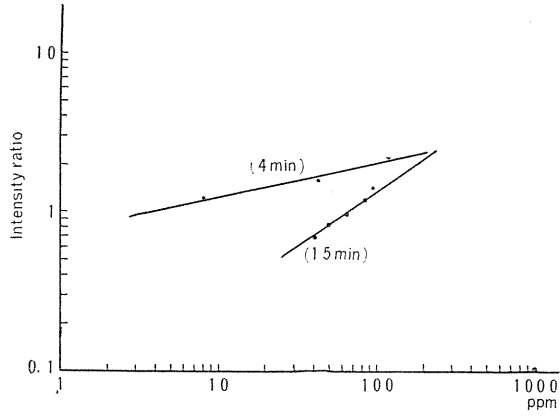
FIG. 3. An example of calibration curve of MgO in BeO.
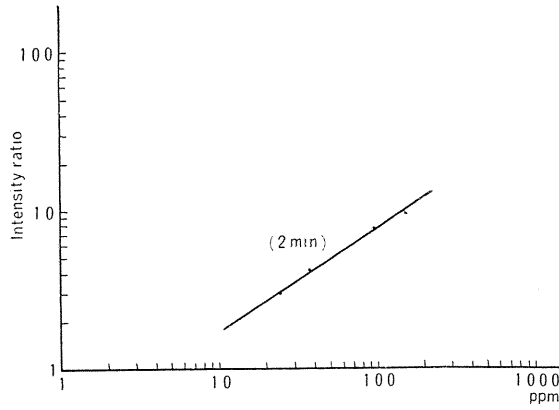

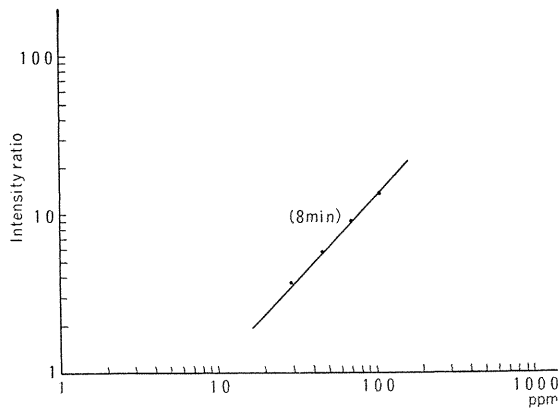FIG. 4. An example of calibration curve of CaO in BeO.

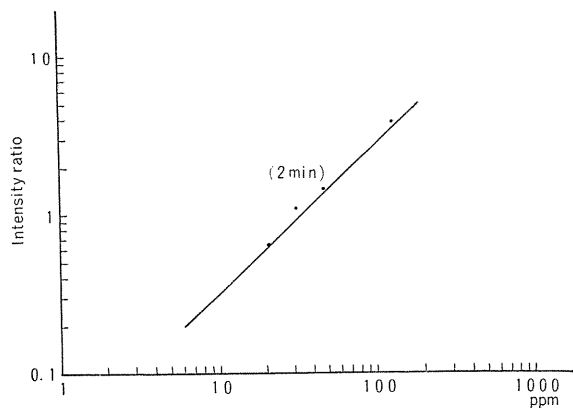FIG. 5. An example of calibration curve of SiO₂ in BeO.

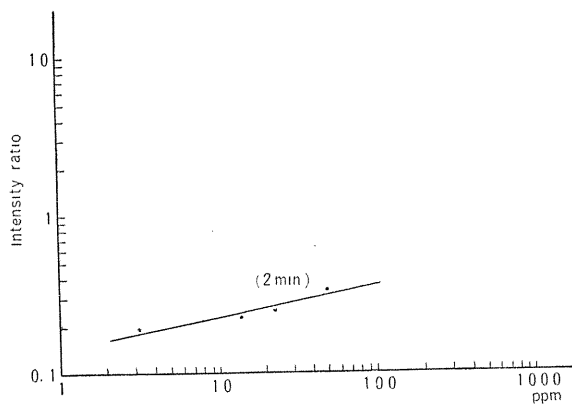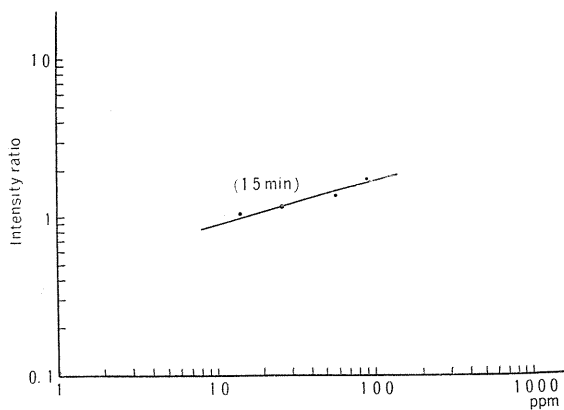FIG. 6.   An example of calibration curve of Fe_2O_3 in BeO.

FIG. 7.   An example of balibration curve of Fe_2O_3 in BeO.

FIG. 8.   An example of calibration curve of MgO in CaO,

Carbon   :   2296.8 Å
(for impurities in beryllium oxide)
Calcium   :   3000.9 Å
(for impurities in calcium oxide or calcium carbonate)

We mixtured a specified amount of calcium oxide or calcium carbonate, silicon dioxide, magnesium oxide and ferric oxide as impurities in beryllium oxide and as impurities in calcium oxide (produced by burning of metallic calcium) we also mixed ferric oxide and magnesium oxide.

4. Experimental Results and Consideration

We investigated four times analytical samples with the above conditions. The experimental results are shown in Table 1. Fig. 3~Fig. 8 are examples of calibration curves in the 1st experiment. Comparing the results with chemical analytical results, we found the results agree with chemical analytical ones.

But it was very difficult to controll the spectrometrical conditions.

TABLE 1. Comparison of Analytical Results

	Impurities in beryllium oxide, (ppm)				Impurities in calcium oxide, (ppm)	
No.	MgO	Fe_2O_3	SiO_2	CaO	MgO	Fe_2O_3
1	11	8	t_r	23	168	36
2	13	10	t_r	22	170	36
3	10	8	t_r	23	170	37
4	11	8	t_r	23	169	36
Chemical analysis	12	8	0	23	170	37

5. Smmary

In this experiment we can conclude as follows;

1. Impurities in beryllium oxide and calcium oxide were determined availably by the condense-spark spectrometric method. (Ferric oxide, silicon dioxide, magnesium oxide and calcium oxide in beryllium oxide, and ferric oxide and magnesium oxide in calcium oxide).

2. Time used for the condense-spark spectrometric method was extremely shorter than that for chemical anaiytical method.

Reference

1) W. Gerlach und E. Riedle: Die chemische Emissionspektralanulyse, Teil III Tabllen zur Quantitativen Analysis (1936).
2) Y. Tachibana and N. Yamaguchi: Shimadzu Review, 10 (1954), 132.