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DETERMINATION OF MICRO AMOUNTS OF IRON, ALUMINUM, AND  
ALKALINE EARTH METALS IN SILICON CARBIDE

H. Hirata and M. Arai

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16. Abstract A colorimetric method for analysis of micro-components in silicon carbide used as the raw material for varistors is described. The micro-components analyzed included iron soluble in hydrochloric acid, iron, aluminum, calcium and magnesium. Standard N.B.S. samples and samples from another company were analyzed by this method, and the results for iron and aluminum agreed well with the N.B.S. standard values and the values obtained by the other company. The method can therefore be applied to the analysis of actual samples.			
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# DETERMINATION OF MICRO AMOUNTS OF IRON, ALUMINUM, AND ALKALINE EARTH METALS IN SILICON CARBIDE

Hiroshi Hirata and Masanao Arai  
Wireless Research Laboratory,  
Matsushita Electric Industrial Co., Ltd., Osaka, Japan.

Micro-components in silicon carbide, the raw material for /820\* varistors, were studied by the method of colorimetric analysis. That is, the iron soluble in hydrochloric acid was determined in the same way as the JIS method by measuring the absorbance of its thiocyanate complex ions at 480 m $\mu$ . Iron and aluminum were also extracted by means of chloroform by generating oxines and chelates at pH 7; they were also determined at the same time by measuring their absorbances at 390 and 470 m $\mu$ . Since the calcium and magnesium contents were very small, their absorbances were measured at 610 m $\mu$  (pH 11.5) using thymolphthalein complexon to obtain their total amount. The values obtained by determining N.B.S. samples with this method agreed extremely well with the standard values. Therefore, the method was applied in the analysis of actual samples, and good results were obtained.

## 1. Introduction

In the manufacturing of silicon carbide, which is used as the chief raw material for varistors, the composition and the amounts of impurities tend to be extremely variable. Since they exert a considerable influence on the electrical characteristics, strict determination of them is a necessity when the raw materials are purchased or used. The JIS R 6124 method [1] is currently available for analyzing silicon carbide. However, this method pertains to silicon carbide used as an abrasive, and it would be

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\* Numbers in the margin indicate pagination in the foreign text.

quite difficult to apply this method unmodified to silicon carbide for varistors, since the impurities contained in it (iron, aluminum, calcium, and magnesium) are all present in quite small amounts.

The authors decided, therefore, to determine all the micro-components by means of colorimetry. The iron soluble in hydrochloric acid was determined by the thiocyanate method; the iron and aluminum were determined by the oxine-chloroform method; and the calcium and magnesium were determined by the thymolphthalein complexon (TPC) method.

## 2. Analytical Methods

### 2.1. Reagents

Iron standard solution (1 mg/ml): 0.2500 g of metallic iron (manufactured by Yokozawa Kagaku, 99.99%) was measured precisely, dissolved in 25 ml of hydrochloric acid (1 + 1), and diluted to 250 ml.

Aluminum standard solution (1 mg/ml): 0.2500 g of metallic aluminum (manufactured by Yokozawa Kagaku, 99.99%) was measured precisely, dissolved in 25 ml of hydrochloric acid (1 + 1), and diluted to 250 ml.

Calcium standard solution (1 mg/ml): 0.6250 g of calcium carbonate (Wakō Junyaku special-grade reagent) was dissolved in 25 ml of hydrochloric acid (1 + 1) and diluted to 250 ml.

Oxine solution: 1.5 g of oxine (Wakō Junyaku special grade) was heated and dissolved in 5 ml of glacial acetic acid and diluted to 250 ml.

Ammonium acetate solution (2M): 15.5 g of ammonium acetate (Wakō Junyaku special grade) was dissolved and diluted in distilled water until the amount reached 200 ml.

TPC solution: 150 mg of TPC (manufactured by Dōjin Yakkagaku) was taken in a 250 ml measuring flask and dissolved and diluted in alcohol.

Triethanolamine solution: 100 ml of distilled water was added to 100 ml of a triethanolamine (Wakō Junyaku special grade) solution.

The sodium carbonate, sodium hydroxide and sodium peroxide used as fusing agents, as well as the other reagents, were all special-grade products of Wakō Junyaku. The indicators used for chelate titration (MX, NN, BT, etc.) were manufactured by Dōjin Yakkagaku.

## 2.2. Equipment

A Hitachi recording spectrophotometer (model EPS-2) was used to measure the absorbances, and a Horiba pH meter (model M-3) was used to measure the pH.

## 2.3. Analytical Procedures

### 2.3.1. Determination of Iron Soluble in Hydrochloric Acid

Exactly 1 g of the sample was measured and taken into a 100 ml beaker. 50 ml of hydrochloric acid (1 + 1) was added to it, and it was covered with a watch glass. It was then gradually heated. After most of the soluble iron had been dissolved, it was left to stand overnight. The insoluble residue was then filtered out and diluted in a 100 or 200 ml measuring flask.

Next the necessary amount was taken into a 50 ml measuring flask, and 5 ml of a 10% ammonium persulfate solution, then 10 ml of a 10% ammonium thiocyanate solution were added to color the iron. Then the absorbance was measured one hour later at 460 mμ, and the iron contents were found with a calibration curve prepared in advance.

### 2.3.2. Decomposition of Silicon Carbide

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Exactly 0.5 g of the sample was measured out and put into a platinum pot together with 5 g of sodium hydroxide and 5 g of potassium nitrate. It was at first heated as slowly as possible to drive out the moisture content thoroughly. Then it was fused completely while maintaining a dark red color. The silicon was removed from the fused substance according to the section entitled "Method for Determining Total Silicon" in 5.2 of the JIS method. This was then used as the test solution for determining the iron, aluminum, calcium and magnesium.

### 2.3.3. Determination of Iron and Aluminum

The silicon which had been separated according to the JIS method was diluted until it reached 200 ml, and 1-10 ml was separated from this diluted filtrate, the exact amount depending upon the amount of iron and aluminum supposed to be present. Then 5 ml of the oxine solution and 5 ml of the ammonium acetate solution were added, the pH was adjusted to 7 by means of aqueous ammonia, and the sample was transferred into a separating funnel and its total amount brought to 100 ml. Next, exactly 10 ml of chloroform was added, the mixture was shaken intensely for 1 minute, and it was then allowed to stand for 5 minutes. Then the chloroform solution was transferred into a small-sized bottle with a ground stopper containing anhydrous sodium sulfate, and its absorbance was measured at 390 and 470 mμ, using as a control

a solution prepared by exactly the same method from fusing on, except that it contained no sample. The iron and aluminum concentrations  $C_{Fe}$  and  $C_{Al}$  were obtained by the following formulas from their respective absorbances  $A_{390}$  and  $A_{470}$ :

$$C_{Fe} = \frac{A_{470}}{a_{470}^{Fe}}, \quad C_{Al} = \frac{A_{390}a_{470}^{Fe} - A_{470}a_{390}^{Fe}}{a_{470}^{Fe} \cdot a_{390}^{Al}}$$

Here,  $a_{390}^{Fe}$ ,  $a_{390}^{Al}$  and  $a_{470}^{Fe}$  are the values of the absorptivity. Incidentally, the values obtained in this way for the iron will contain also the iron soluble in hydrochloric acid, and it will be necessary to subtract this amount.

### 2.3.4. Determination of Alkaline Earth Metals

The silicon was separated according to the JIS method, and 10 ml was taken from the filtrate which had been diluted to 200 ml. Then 1 ml of a 50% triethanolamine solution and 1 ml of a 10% ammonium chloride solution were added to this. Next 5 ml of a 0.06% alcohol solution of TPC and 25 ml of concentrated aqueous ammonia were added, and the solution was diluted in a 50 ml measuring flask. Its absorbance was then measured at 610 mμ, using the test solution as a control, since the latter contains no sample. The alkaline earth metal contents were then found with a calibration curve prepared in advance in the same way.

## 3. Study of the Analytical Methods

### 3.1. Study of the Method for Determining Iron Soluble in Hydrochloric Acid

The JIS mentions two methods for determining the iron which becomes mixed with the silicon carbide during the crushing process: the potassium permanganate titration method and the colorimetric method using potassium thiocyanate. In this case we used the

latter colorimetric method. However, the thiocyanate complex ions will fade rapidly if the JIS method is used without modification, and we therefore made a study of the method of adding ammonium persulfate [2]. The results are as shown in Fig. 1.

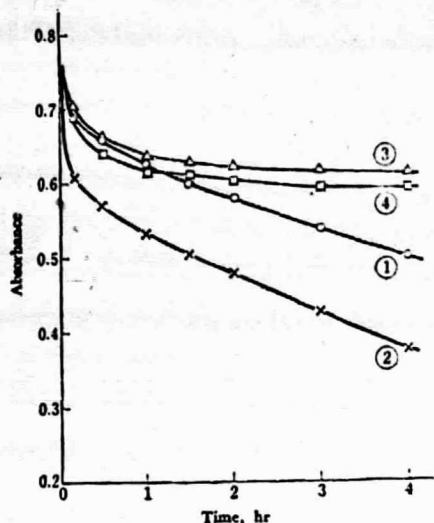


Fig. 1 Effect of standing time on absorbance of ferric thiocyanate complex

① 1%  $\text{NH}_4\text{SCN}$  in 1.2N  $\text{H}_2\text{SO}_4$ , ② 1%  $\text{NH}_4\text{SCN}$  in 1.2N  $\text{HCl}$ , ③ 1%  $\text{NH}_4\text{SCN} + 1\%$   $(\text{NH}_4)_2\text{S}_2\text{O}_8$  in 1.2N  $\text{H}_2\text{SO}_4$ , ④ 1%  $\text{NH}_4\text{SCN} + 1\%$   $(\text{NH}_4)_2\text{S}_2\text{O}_8$  in 1.2N  $\text{HCl}$

(1) and (2) in Fig. 1 show the measured values of the absorbances of ferric chloride and ferric sulfate prepared in the following manner. Amounts of each were taken in 100 ml measuring flasks, and 10 ml of 12N hydrochloric acid or 12N sulfuric acid was added to them. Then 10 ml of a 10% ammonium thiocyanate solution was added, and the solutions were diluted with distilled water until they each came to 100 ml. Then their absorbances were measured with the passage of time (hours). In the

samples in curves (3) and (4), 10 ml of a 10% ammonium persulfate solution was added before the addition of the ammonium thiocyanate.

As is clear in Fig. 1, the absorbance declines remarkably with the passage of time in samples which contain no ammonium persulfate, no matter whether they contain hydrochloric or sulfuric acid. However, if ammonium persulfate is added, their absorbance will become more or less constant after a certain time. Therefore, the measurements were taken about 1 hour after the addition of ammonium persulfate and ammonium thiocyanate. Incidentally, the sulfur will be liberated rapidly if ammonium thiocyanate and ammonium persulfate are used in excessive amounts, and the limit is 10 ml in a 50 ml measuring flask. The absorption

maximum wavelengths will also differ depending upon the amounts of these reagents which are added, but 480 mμ is believed to be best.

### 3.2. Study of Method for Decomposing Silicon Carbide

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In the study we followed the JIS fusing method, in which 10 g of sodium carbonate or sodium hydroxide and 4 g of potassium nitrate are added to 0.5 g of the sample, which is then fused in a nickel pot while maintaining a dark red color. However, the silicon carbide to be determined here can be fused by the sodium carbonate-potassium nitrate method only with difficulty. If an elevated temperature is adopted, the fusing agent will spatter or will flow out along the walls of the pot, and it is therefore necessary to repeat the process a number of times at a low temperature. In fact, there are some samples which cannot be fused even when the process is repeated. However, if the sodium hydroxide-potassium nitrate method is carried out in the manner described in section 2.3.2, fusion will be completed in about 1 hour. In this case, there will be good fusion at a low temperature even if the sample and fusing agent are not thoroughly mixed together. In addition to the JIS method described above, we also made a study of the sodium carbonate-sodium peroxide method in the interests of speeding up the fusion process. We found that if the heat is increased somewhat the reaction with the sample will proceed at an explosive rate and the fusion will be completed within a short time. However, large amounts of nickel will be eluted, and there will also be danger that the sample and fusing agent may spatter. This, this method is lacking in reproducibility.

Consequently, the JIS sodium hydroxide-potassium nitrate method was adopted on account of the circumstances described above. The amounts of fusing agents were 5 g of sodium hydroxide and 5 g of potassium nitrate for every 0.5 g of sample. Since

the amounts of nickel from the pot which are mixed into the sample are variable, nickel pots were deemed to be unsuitable for analyzing iron and aluminum, and it was decided to use platinum pots in this case.

### 3.3. Study of Method for Determining Iron and Aluminum

In the JIS method, both iron and aluminum are precipitated simultaneously as hydroxides, and they are both weighed as oxides. Next, the iron contents are found by the potassium thiocyanate method, and the amount of aluminum is calculated by subtraction. However, the samples analyzed here contain far less iron and aluminum than the amounts contained in silicon carbide used as an abrasive. Therefore, if the JIS method is adopted, not only will the error based on the method itself be increased, but it will also be necessary to fuse large amounts of the sample with large amounts of fusing agents, so that iron and aluminum impurities originating in the fusing agents and other sources may very well be mixed in the samples. For this reason, in this case we studied a method of extracting the iron and aluminum in chloroform in the form of oxine salts and performing the colorimetric analysis at the same time [3].

#### 3.3.1. Absorption Curves

The oxine salt of iron in a chloroform solution has its absorption maxima at 370, 470 and 580 m $\mu$ , as is shown in (A) of Fig. 2. On the other hand, the oxine-chloroform extract of aluminum has absorption at 390 m $\mu$ , but it has almost none at wavelengths greater than 450 m $\mu$ , as is shown in (B). In addition, the absorption of iron at 390 and 470 m $\mu$  and that of aluminum at 390 m $\mu$  both follow Beer's law quite well, as is shown in Fig. 3. Therefore, it would be possible to determine both iron and aluminum simultaneously in accordance with the formula in 2.3.3.

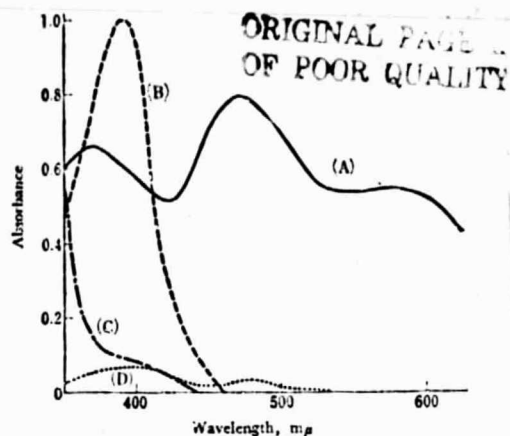


Fig. 2 Absorption spectra

(A) Fe-Oxine in chloroform, Reference: Oxine in chloroform; (B) Al-Oxine in chloroform, Reference: Oxine in chloroform; (C) Oxine in chloroform, Reference: Chloroform; (D) Fusing agent-Oxine in chloroform, Reference: Oxine in chloroform

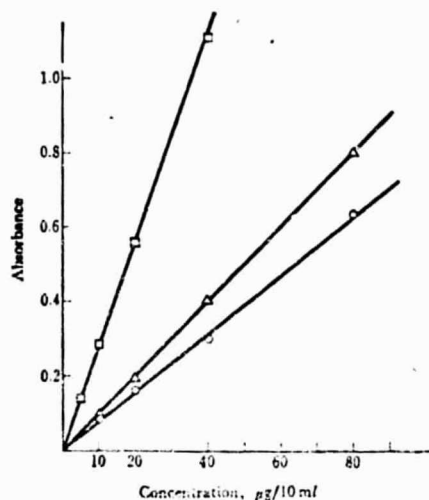


Fig. 3 Calibration curves of aluminum and iron

—□—  $\text{Al}_{390}$ , —△—  $\text{Fe}_{470}$ , —○—  $\text{Fe}_{330}$

Nevertheless, since the absorption curves in blank tests showed considerable absorption at 390 and 470  $\text{m}\mu$ , as is shown in (D) of Fig. 2, it was decided to use a control solution for the actual samples in the actual analysis. The control solution was prepared exactly in the same way as the sample solutions, with the sole difference that it contained no sample.

### 3.3.2. Influence of pH during /823 Extraction

We investigated the pH when the oxine salts of iron, aluminum and the control solution were quantitatively extracted in chloroform. It was found as a result that the pH of iron was 2.4 or higher and that of aluminum was 4.5 - 9.5. When the absorbance of the control solution was being measured at 390  $\text{m}\mu$ , it became constant at 4.5 - 9.5. Consequently, it will be sufficient to carry

out the extraction at a pH of 4.5 - 9.5. However, when a study was made with actual samples, it was found as a result that a pH of 6 - 8 was better than one of 5, as is shown in Table I. For this reason, it was decided to use a pH of 7 during extraction.

Table I Effect of pH on extraction of oxine salts of iron and aluminum with chloroform

pH	$A_{390}$	$A_{470}$
5	$0.905 \pm 0.115$	$0.545 \pm 0.015$
6 - 8	$1.028 \pm 0.027$	$0.553 \pm 0.007$

### 3.3.3. Influence of Impurities in Fusing Agents

Large amounts of fusing agents are used in fusing silicon carbide. The fusing agents (special-grade reagents) contain considerable amounts of iron, aluminum and other elements which might interfere with measurement of the absorbance. Should there be pronounced differences in the impurities depending on the time and place of collection of samples, considerable errors might possibly result on account of the minute amounts of iron and aluminum contained in the samples themselves. In order to find out the degree to which this is the case, we followed the actual analytical procedures to find the absorbance of the fusing agents at 390 and 470 mμ, using an oxine-chloroform solution as the control. The values are shown in Table II. These results show that the fluctuations in absorbance caused by the fusing agents are very slight and that, even if a platinum crucible is used, the disturbances caused by elements eluted from the crucible are quite negligible.

Table II Absorbance of fusing agent extracted  
by oxine-chloroform

Sample	$A_{390}$	$A_{470}$
Fusing agent (I)	$0.074 \pm 0.011$	$0.032 \pm 0.006$
" (II)	$0.074 \pm 0.011$	$0.031 \pm 0.005$

(I) : 5g NaOH+5g KNO<sub>3</sub>, (II) : (I) Melted in platinum crucible

### 3.3.4. Influence of Other Ions Present

Silicon carbide was given quantitative analysis by emission spectrometry, and the results showed that, in addition to iron and aluminum, other elements such as copper, manganese, calcium, magnesium and strontium were also present. Of these, copper would obviously disturb the analysis, but its concentration was small and negligible. Manganese also would be difficult to extract at this pH, but its concentration was also very small, and it poses hardly any problem. The alkaline earth metals were also found to be completely negligible. Thus, it was concluded that there is no need to consider the influence of other elements which may be present.

Table III Absorbance and absorption coefficient  
of oxine salts of iron and aluminum  
in chloroform

Amount added ( $\mu\text{g}/10\text{ ml}$ )	$A_{390}^{\text{Fe}}$	$\epsilon_{390}^{\text{Fe}}$	$A_{470}^{\text{Fe}}$	$\epsilon_{470}^{\text{Fe}}$	$A_{390}^{\text{Al}}$	$\epsilon_{390}^{\text{Al}}$
5.0	—	—	—	—	0.141	0.0282
10.0	0.082	0.0082	0.098	0.0098	0.287	0.0287
20.0	0.148	0.0074	0.193	0.0097	0.538	0.0279
40.0	0.300	0.0075	0.402	0.0101	1.117	0.0279
80.0	0.633	0.0079	0.801	0.0100	—	—
Av.	—	0.0078	—	0.0099	—	0.0282

Reference : Reagen blank

Table IV Analytical results for determination of  
iron and aluminum in synthetic solution

	10 ml/200 ml taken				1 ml/200 ml taken			
	Amount of iron ( $\mu\text{g}/\text{ml}$ )		Amount of aluminum ( $\mu\text{g}/\text{ml}$ )		Amount of iron ( $\mu\text{g}/\text{ml}$ )		Amount of aluminum ( $\mu\text{g}/\text{ml}$ )	
	added	found	added	found	added	found	added	found
A	50.0	51.3	50.0	49.8	50.0	49.1	50.0	49.6
B	50.0	49.6	2.5	1.7	50.0	47.6	2.0	1.2
C	5.0	5.8	50.0	49.6	5.0	5.9	50.0	50.0
D	5.0	4.8	2.5	2.4	5.0	4.5	2.0	2.1

Table V Analytical results of synthetic solution

10 ml/200 ml taken				1 ml/200 ml taken			
Fe added	Fe found	Al added	Al found	Fe added	Fe found	Al added	Al found
2%	—	2%	—	2%	1.981%	2%	1.801%
0.2	0.198	0.2	0.194	0.2	0.203	0.2	0.183
0.02	0.026	0.02	0.021	0.02	—	0.02	—

### 3.3.5. Studies with Synthetic Samples

On the basis of the results of the studies mentioned above, we prepared the calibration curves and found the absorption coefficients. The method used was the same as that described in section 2.3.3, with the difference that the prescribed amounts of iron and aluminum were added separately to solutions in which the fusing agents had been processed in the same way as the test solutions (2.3.2).

A study was made with synthetic samples to determine the degree of accuracy of the calibration curves of Fig. 3, which were obtained in this way, and the absorption coefficients of Table

III. The analytical results are shown in Tables IV and V. The results shown in Table IV were for four samples when the amounts taken were 10 ml/200 ml and 1 ml/200 ml. At this range of concentrations, the amount of sample taken makes no difference. When there is a large amount of iron and a small amount of aluminum, 824 the analytical value for aluminum will tend to be small. On the contrary, when there is a large amount of aluminum and a small amount of iron, the analytical value for iron will tend to be large. Table V shows the results when the amounts of iron and aluminum added were 10, 1 and 0.1 mg each. When the percentages ranged from 2% to about 0.2%, an amount of 1 ml/200 ml was sufficient, but when the percentage was less than 0.2%, it was necessary to take 10 ml/200 ml.

#### 3.4. Study of Method for Determining Alkaline Earth Metals

JIS adopts a method in which calcium is precipitated as an oxalate and magnesium as magnesium-ammonium phosphate. After they have each been filtered out, they are exposed to a strong heat and weighed. However, this method is applicable only in cases where they are present in rather large concentrations, and some other method must be considered for silicon carbide used as an electronic material. One such method would be flame spectrophotometry, but it would be difficult to apply because of the large amounts of sodium and potassium in the test solutions. The EDTA titration method is beyond the range where analysis is possible. It might be possible to use emission spectroscopy, but in this case we studied the method of colorimetry with the indicators used in EDTA titration. Of the indicators for calcium and magnesium, we made studies of NN, BT, MX and TPC [4]. We found as a result that TPC had the greatest absorbance for calcium concentrations on the order found in the actual samples; it also had the best stability. Therefore, in the study described below, we limited ourselves to TPC alone and made a study of calcium.

##### 3.4.1. Amounts of TPC Added and pH

It was found that if TPC were added in an amount of 2,000  $\mu\text{g}/50\text{ ml}$  to calcium in amounts of 100  $\mu\text{g}/50\text{ ml}$ , this would be quite sufficient for the production of chelates. Furthermore, a pH of 10 - 12 is sufficient for EDTA titration, but it is questionable whether this is suitable for colorimetry. A study was made of this, and it was found that the absorbance reached its maximum and become constant at a pH of 11 - 12. Therefore, a pH of 11.5 was used in the following.

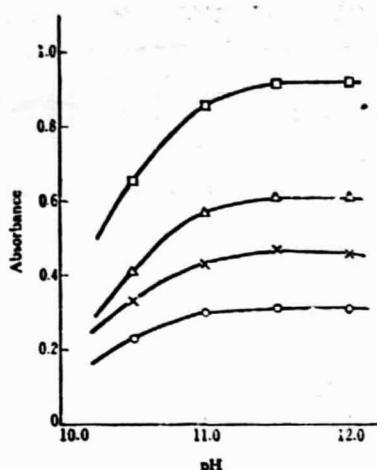


Fig. 4 Effect of pH on absorbance of Ca-IPC chelate compound

Ca (µg/ml): —□— 1.6, —△— 0.8, —x— 0.4, —○— 0

Table VI Determination of calcium in synthetic solution

Added	Sample (mg)			
	A	B	C	D
NaOH	5000	5000	5000	5000
KNO <sub>3</sub>	5000	5000	5000	5000
Ca		0.5	0.5	0.05
Al			2.5	2.5
Fe			2.5	2.5
Cu			0.05	0.05
Mn			0.15	0.15
Ca found	0.36	0.45	0.47	0.07

Reference of A : Reagent blank; Reference of B, C and D : A

### 3.4.2. Influence of Other Ions Present

As was mentioned in 3.3.4, small amounts of iron, aluminum, copper, manganese, strontium, calcium and magnesium are contained in silicon carbide. Of these, the copper and manganese concentrations are extremely small, and they are negligible. However, it is believed that the iron (0.5 - 0.05%) may have a considerable influence, and it is detected with almost the same sensitivity as the magnesium (0.03 - 0.003%) and strontium (0.01 - 0.001%). By adding triethanolamine, we were able to mask the iron almost perfectly. However, since the alkaline earth metals are difficult to separate, it was decided to find their total amounts.

### 3.4.3. Studies with Synthetic Samples

On the basis of the results of the studies outlined above, we prepared the synthetic samples shown in Table VI and made studies of the operating procedures. A study of the measured values in Table VI shows that the control solutions had a quite great absorbance. Consequently, if the concentrations are very small, rather large errors will result. This indicates that rather large amounts of alkaline earth metals and other disturbing elements are present in the fusing agents and other reagents used.

The elimination of the disturbing elements contained in the reagents and distilled water, as well as the methods themselves must be studied with greater strictness.

#### 4. Determination of Actual Samples

Standard samples (N.B.S.) and samples (manufactured by company N) were analyzed according to the procedures described in section 2.3, and the results were compared with the N.B.S. standard values and with the values determined by company N (using a completely different analytical method). The results are shown in Table VII.

Table VII Comparison of present method and the other method for determination of iron, aluminum and alkaline earth metals in silicon carbide

Sample	Method	Fe	Al	Ca + Mg	Dis. Fe†
NBS	Standard	0.45	0.23	0.05	—
	M	0.47	0.24	0.07	0.12
	N	0.42	0.23	—	—
SiC-A	M	0.24	0.57	0.07	0.14
	N	0.22	0.56	—	—
SiC-B	M	0.13	0.09	0.03	0.05
	N	0.17	0.10	—	—

Standard: Method of NBS, M: Present method, N: Other method performed by N. Co. Ltd.

† Dis. Fe: Iron mixed up during the crushing process of silicon carbide. It was dissolved in hydrochloric acid solution.

The analytical values for the iron soluble in hydrochloric acid could not be evaluated for their 825 accuracy since there are no reference values with which they can be compared. The analytical values for the calcium and magnesium totaled together were somewhat greater than the N.B.S. standard values. This is because unsolved

problems are still present in the analytical method, as was mentioned before, and we must await further studies in the future. However, the results for iron and aluminum agreed well with the N.B.S. standard values and the analytical values obtained by company N, and it was learned that this method can be applied to the analysis of actual samples.

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