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# DETERMINATION OF THE PURE SILICON MONOCARBIDE CONTENT OF SILICON CARBIDE AND PRODUCTS BASED ON SILICON CARBIDE

L. Prost and A. Pauillac

Translation of "Contribution à la détermination de la teneur en SiC pur du carbure de silicium et des produits à base de carbure de silicium," Bulletin de la Société Française de Céramique, No. 84, 1969, pp. 75-83.

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**16. Abstract** Experience has shown that different methods of analysis of SiC products give different results. Three methods identified as AFNOR, FEPA , and manufacturer P, currently used to det. SiC, free C, free Si, free Fe, and $SiO_2$ are reviewed. The AFNOR method gives lower SiC content, attributed to destruction of SiC by grinding. Two products sent to independent labs. for anal. by the AFNOR and FEPA methods showed somewhat different results, especially for SiC, $SiO_2$, and $Al_2O_3$ content, whereas an x-ray analysis showed a SiC content approximately 10 points lower than by chemical methods. The cause of the different results produced by grinding has not been studied.

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# DETERMINATION OF THE PURE SILICON MONOCARBIDE CONTENT OF SILICON CARBIDE AND PRODUCTS BASED ON SILICON CARBIDE

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Experience shows that the different methods usually used for determining content of pure SiC either in silicon carbide or in products based on silicon carbide, lead to large errors. /75*

This information, as precise as possible, is, however, indispensible to all technical progress.

This study describes summarily, in the first part, three methods for analysis currently used; then in the second part, their application for two types of products is described.

Finally, the comparison of the results obtained on silicon carbide and the products containing it has made it possible to choose analysis methods from each of these which assure the best agreement.

## 1. Chemical Analysis of Silicon Carbide

### 1.1. Preparation of a Sample for Titration of SiC.

### 1.1.1. The AFNOR [Association francaise de normalisation, French Association of Standards] Method.

The sample, ground in a steel mortar, passes through a mesh sieve with squares 0.125 mm--iron is removed by a magnet. Sample of 2 g.

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

### 1.1.2. A Method of Chemical Analysis of Impurities from the Surface of Silicon Carbide for Abrasive Products of FEPA [Fédération Européenne des Produits Abrasifs, European Federation of Abrasive Products] Proposal.

The product to be analysed must pass through a mesh sieve with squares 3.36 mm (No. 6 ASTM).

If necessary, the rejects are ground totally in a steel mortar in order to pass through a 3.36 mm mesh. Any grinding which is finer must not be done.

Before weighing, it is dried 1 hour at 110° C.

In the case of classified grains, weighing is done directly for analysis after thorough mixing of the samples.

In the case of grains which all go through, they are screened according to fractions 3.36/0.707 mm--0.707/0.250 mm--finer than 0.250 mm. Each fraction is weighed.

The 5 to 10 g sample for analysis will be reconstituted by weight according to each class of the aforementioned granules.

### 1.1.3. Method Used by Producer P of Silicon Carbide

The ground sample passes through mesh No. 120 (0.124 mm). Sample of 2 g.

### 1.2. Titration of SiC /76

### 1.2.1. AFNOR Method

Attack on the 2 g sample in a platinum capsule by 20 mℓ of HF + a few drops of $H_2SO_4$ d = 1.83, is evaporated till dry without boiling; eliminating the white fumes of $SO_3$ by heating is most important without, however, bringing the capsule to red.

Repetition with 20 mℓ of HCl d = 1.19 in the capsule, bringing it near the boiling point for 30 min. One brings it to 150 mℓ by adding distilled water in a porcelain capsule. It is filtered through a common filter, washed in boiling water until the Cl ions disappear, bringing the filtrate to 250 mℓ.

A dried residue is obtained with its filter at 110° C for 30 min and following this it is roasted at 800° C for 15 min.

The calcined residue is dried and gives $SiC = (M - b) \frac{100}{E}$

M = the mass of SiC
*b* = blank test
E = sample

A blank test is done in parallel (= *b*).

The standard indicates, in effect, carrying out an oxidizing attack when the amount of SiC is uncertain.

### 1.2.2. Method Proposed by FEPA

The sample of 5 to 10 g is attacked in a 50 mℓ platinum crucible by 15 mℓ HF + 2 or 3 drops of $H_2SO_4$. The solution is evaporated until dry at a moderate temperature for less than 6 hours, is mixed from time to time with a platinum wire. Then, the temperature is raised slightly until the sulfur trioxide fumes are clearly visible, and heating is continued for another 10 min.

Repetition with 25 mℓ of HCl 1/1 and one or two drops of $H_2SO_4$ at 98% in a crucible, heating it to near the boiling point for 30 min mixing from time to time in order to disengage the sample from the crucible.

The residue is transferred to a calcined glass filter with porosity No. 4, is washed and rinsed completely in hot water.

The calcined glass filter is dried at a constant load between 120 and 130° C.

The residue is dried, it gives: SiC + C + Si

$$\text{Calculation of the \% of SiC} = \frac{P_2}{P_1} \times 100 - C\% - Si\%$$

$P_1$ = weight of the sample
$P_2$ = weight of the filter and dried product
C% = free carbon, percent
Si% = free silicon, percent

The sample is saved for titration of free silicon and free carbon.

The SiC will be obtained by the difference, after analysis, of Si and C.

### 1.2.3. Method Used by Producer P

It is identical to the AFNOR method.

## 1.3. Titration of Free Carbon

### 1.3.1. AFNOR Method

The 3 g sample taken passing 0.125 mm, is in a porcelain capsule 60 mm in length and 35 mm in width.

Introduced into an electric muffle furnace, with the atmosphere slightly oxidizing, it is calcined at 700--750° C for 1 hour, effecting calcining lasting 5 min until the weights following the last 3 calcine treatments indicate a constancy of the mass.

The difference of weight before and after calcining gives free carbon

$$C\% = (M_1 - M)\frac{100}{E}$$

$M_1$ = the mass of the capsule and the sample

M = the mass of the capsule and the residue after calcining

E = the sample

### 1.3.2. Method Proposed by FEPA

Three different apparatuses are used for determining free carbon.

The first two use an electric furnace. They differ in the /77 position of the furnace, in the first case the furnace is horizontal, in the second, it is vertical.

The following equipment units are mounted in series:

1) source of oxygen
2) flow regulator
3) washing flask containing $H_2SO_4$
4) drying column containing $Mg(ClO_4)_2$
5) column for absorption of $CO_2$
6) horizontal or vertical furnace (with a capsule or crucible)
7) column containing granulated zinc
8) column containing $P_2O_5$
9) column for $Mg(ClO_4)_2$
10) an absorber containing a $CO_2$ absorbant (Ascarite)

The third uses a high-frequency induction furnace with the following units mounted in series:

1) source of oxygen
2) flowmeter

3) washing flask containing $H_2SO_4$
4) column containing $Mg(ClO_4)_2$ anhydrous
5) absorption column of $CO_2$ (Ascarite)
6) high-frequency induction furnace (with crucible)
7) tube containing a sulfur absorber
8) a furnace for catalysis to transform CO to $CO_2$
9) column containing $P_2O_5$
10) column for $Mg(ClO_4)_2$ anhydrous
11) absorber containing a $CO_2$ absorbant (Ascarite)

The method of operation for the three apparatuses consists of making, first, a blank test sending a current of oxygen with a flow of 300 mℓ/min for 15 min, the furnace having a temperature of 900--950° C. The absorber is weighed after the oxygen is stopped.

After having cooled the scoop, 1 to 2 g of the sample is introduced, placing the scoop in a heated zone, connecting the absorber and immediately sending oxygen with a flow of 300 mℓ/min, maintaining the current for 15 min at a furnace temperature of 900--950° C, withdrawing the absorber and the scoop, cooling in a dessicator, and weighing the cold absorber as well as the sample.

Calculation: free carbon $= \dfrac{0{,}17\,P_5 + 0{,}37\,(P_3 - P_4)\,\%}{P_3\,f}$

$P_3$ = weight of the sample before burning
$P_4$ = weight of the sample after burning
$P_5$ = weight of $CO_2$ absorbed
$f = \dfrac{P_1}{P_2}$ (correction as a result of loss of weight by hydrofluoric corrosion--see titration of SiC).

### 1.3.3. Method Used by Producer P

A 3 g sample, passing through a 120 screen, is in a porcelain capsule 60 mm in length and 35 mm in width, tare $T_1$.

It is introduced to a muffle furnace at 800° C, remaining 1 min, is brought out and let cool, weighed to tare $T_2$; the scoop is introduced again into the furnace for 2 min, let cool to tare $T_3$; calcining is begun again for 2 min, tare $T_4$; calcinings are continued for a duration of 5 min up until the weights of the last three calcinings indicate an increase in weight contained in the scoop.

The tares $T_1$, $T_2$, $T_3$, $T_4$, etc., make it possible to construct a curve of the variation of weights of the scoop as a function of duration of calcining.

One holds at the abscissa, successive times and on the ordinate, the corresponding weights. Following the curve, one deduces the $T_m$ tare corresponding to loss of maximum weight.

$T_m - T_1 = P =$ weight of calcined carbon
$E =$ the sample in grams $= 3$
$P \times \frac{100}{E} = C\%$

## 1.4. Titration of Free Silicon

### 1.4.1. AFNOR Method

The sample: 2 g of ground sample passing through a mesh sieve with squares 0.40 mm (E).

One carries out a blank test in identical fashion (b).

It is attacked in a platinum capsule by 25 mℓ of a solution of silver fluoride at 20 g/ℓ and 3 mℓ of hydrofluoric acid d = 1.14 brought to 70° C for 1 hour with the silver precipitate being stirred every 5 min, and filtered on a flat filter for rapid filtration, the filtrate going into a beaker with paraffin. It is washed 5 times in hot water, the precipitate dissolved on a filter by 25 mℓ of a solution of nitric acid at 1/2, washed 5 /78 times in hot water, and cooled. Two to three drops of a solution of 200 g/ℓ of iron alum and ammonia is added; it is titrated with a N/10 liquid of thiocyanate of ammonia until there is a pink coloration, or (N) mℓ of the volume is used. The ammonia thiocyanate liquid has been previously titrated with a solution of silver nitrate N/10, being (f) the correction factor of the solution of ammonia thiocyanate.

Calculation:

a)

$$(\text{free Si} + 0.25 \text{ free Fe})\% = (N - b) \times t \times f \times \frac{100}{E}$$

$t$ = theoretical titer = number of grams of silicon for 1 mℓ of N/10 liquid of ammonia thiocyanate ($t$ = 0.000707)

b)

$$(\text{free Si} + \text{free } 0.25 \text{ Fe})\% - (0.25 \text{ free Fe})\% = \text{free Si }\%.$$

## 1.4.2. Method Proposed by FEPA

Sample (P) = 2 to 4 g of silicon carbide residue previously attacked by HF + $H_2SO_4$ (see titration of SiC).

It is attacked in a polyethylene beaker by 15 mℓ of a solution of silver fluoride prepared at least 24 hours in advance according to:

10 g of silver monoxide in 500 g HF at 40% is left in contact for 2 hours and mixed from time to time to filter on paper and a polyethylene funnel, is washed with hot water until cloudiness disappears with the addition of HCl.

The sample with its filter is transferred into a 250 mℓ Erlenmeyer flask.

About 50 mℓ of nitric acid 1/1 is added and heated to boiling.

It is titrated using a solution of (V) mℓ of ammonia thiocyanate N/10 (normal N) verified beginning with a decinormal solution of silver nitrate, using a few drops of iron alum and ammonia as the indicator, solution 200 g/ℓ.

$$\text{Calculation:} \quad \text{free Si} = \frac{V \times 0.702 \times N}{P \times \frac{P_1}{P_2}}$$

$P_1$ = weight of the sample before sulfo-hydrofluoric attack

$P_2$ = weight after sulfo-hydrofluoric attack (see titration of SiC).

### 1.4.3. Method Used by Producer P

It is identical to the AFNOR method.

## 1.5. Titration of Free Iron

### 1.5.1. AFNOR Method

The sample equals 25 g of non-ground sample.

It is attacked in a 250 mℓ Erlenmeyer flask with 50 mℓ of water and 60 mℓ of $H_2SO_4$ d=1.83; it is brought to boiling for 30 minutes, diluted to 200 mℓ, filtered in a vacuum. It is washed five times in cold water, increased to 500 $cm^3$ with distilled water, and 5 mℓ of nitric acid is added d=1.33, it is brought to boiling for 10 min,and increased to 1000 $cm^3$ with distilled water.

Colorimetric titration in identical glass tubes containing 10 $cm^3$ of solution (V) compared to a range of standard samples carried out beginning with the solution of ferric sulfate at 0.025% of iron; indicator is 1 mℓ of ammonia thiocyanate N/10.

### 1.5.2. Method Proposed by FEPA

Volumetric titration.

Metal oxides of the filtrate coming from sulfo-hydrofluoric attack of silicon carbide are precipitated, filtered, calcined, and then submitted to fusion by potassium pyrosulfate; it is repeated with $H_2SO_4$ or an HCl, the sulfur is precipitated from them by a current of $H_2S$, the filtrate which contains the iron is reduced by stannous chloride, and the excess of stannous chloride is oxidized by mercuric chloride.

The ferrous salt is titrated by bichromate of potassium with diphenylamine sulfonate of barium.

Colorimetric titration. /79

Putting into solution the metallic oxide resulting from the sulfo-hydrofluoric attack is the same as for volumetric titration. The colorimetry is effected in the presence of standard samples with hydroxylamine hydrochloride, orthophenantroline and ammonia acetate. A blank test is accomplished.

The optical density is measured at 510 millimicrons.

### 1.5.3. Method Used by Producer P

It is identical to the AFNOR method.

## 1.6. Titration of Free Silica

### 1.6.1. AFNOR Method Test

The 2 g sample (F) passes through a mesh sieve with squares of 0.125 mm.

It is carried out in identical fashion to the blank test (b).

The attack is in a platinum capsule with 20 mℓ of HF at 40% and a few drops of $SO_4H_2$ d=1.83; it is evaporated till dry without boiling, the excess of sulfuric acid is expelled on a small flame, calcining at 800° C to a constant mass.

$$\text{Calculation:} \quad (M - b) \times \frac{100}{E}$$

M = mass of free Si + free C

Free $SiO_2$% = (free $SiO_2$ + free C)% - free C%.

### 1.6.2. Method Proposed by FEPA

Beginning with sulfo-hydrofluoric attack for titration of SiC already accomplished, we have:

$P_1$ -- is the weight of the sample,

$P_2$ -- is the weight of the dry residue filtered and dried,

$V_1$ -- is the total volume of filtrate obtained.

An aliquot intake of $V_2$, 100 $cm^3$ of volume $V_1$ is introduced in a platinum capsule and evaporated till dry until the end of $SO_3$ vapor emission. The residue is calcined in an electric furnace to constant weight $P_6$.

$$\text{Calculation:} \quad \text{free } SiO_2\% = \frac{P_1 - P_2 - P_6 \frac{V_1}{V_2}}{P_1} \times 100\%.$$

If the sample contains calcium or magnesium, the weight of the residue $P_6$ will be corrected for corresponding weight $SO_3$ contained in $CaSO_4$ and $MgSO_4$.

### 1.6.3. Method Used by Producer P

It is identical to the AFNOR method.

## 1.7. Titration of metallic oxides

The methods used for titrating $R_2O_3$, CaO, MgO, $Na_2O$, $K_2O$, enter into the domain of classical analysis which will be not discussed.

# 2. Refractory Products on a Base of Silicon Carbide

The analysis methods are similar to those variants used for analysis of silicon carbide.

# 3. Experimental Study of Determination of the Content of SiC in Technical Silicon Carbide

Two samples, E1 and E2, have been subjected to different analysis methods by two laboratories, A and B, in order to determine, principally, the content of SiC and $SiO_2$, the other constituents are not titrated except for verification titers.

## 3.1. E1, 0-2 mm All Inclusive Following AFNOR Grinding to 125 Microns is Attacked by Sulfo-Hydrofluoric or an Oxidizing Agent.

One recalls that an oxidizing attack is made according to sample of 0.250 g of technical SiC ground to 125 microns with oxidizing melting in a crucible made of nickel heated to 500°C.

/80

| | Laboratory A | | Laboratory B | |
|---|---|---|---|---|
| | Sulfo-hydrofluoric attack | | Sulfo-hydrofluoric attack | Oxidizing attack |
| SiC | 84.40 | | 82.42 | 80.00 |
| $SiO_2$ | 10.40 | | | 10.20 |
| C free | 0.20 | | 10.95 | 0.20 |
| $Al_2O_3$ | 1.28 | ) | | |
| $TiO_2$ | traces | ) | | |
| $Fe_2O_3$ | 0.32 | ) | | |
| CaO | 0.80 | )on the filtrate | Non titrated | Non titrated |
| MgO | 0.29 | ) | | |
| $Na_2O$ | 0.26 | ) | | |
| $K_2O$ | 0.40 | ) | | |

There was breakdown of SiC into Si and C which combine, respectively, with the oxygen in order to produce $CO_2$ and $SiO_2$, The coefficient of transformation of Si to $SiO_2$ is equal to 1.5.

In the case above, the quantity of total $SiO_2$ found was equal to 130.2 from which it is convenient to subtract the free silica which exists already in the product which one other sulfo-hydrofluoric attack has determined, in another way, to equal 10.20, so that:

$$130.20 - 10.20 = 120$$
$$\text{from which SiC} = \frac{120}{1.5} = 80.00$$

### 3.2. On E2, 0-2 mm All Inclusive.

### 3.2.1 According to the AFNOR Method -- Grinding to 125 Microns

The analyses concerning SiC and $SiO_2$ carried out by laboratory A are the following:

Sulfo-hydrofluoric attack:

SiC = 82.00

Free $SiO_2$ = 12.20

Oxidizing attack:

Total $SiO_2$ = 130.00

$$SiC = \frac{130 - 12.20}{1.5} = 78.50$$

3.2.2. The methods of attack according to AFNOR are the same as in 3.2.1, but technical silicon carbide is not ground, taken in such a way that it is all 0-2 mm and is separated into 3 fractions in which granulometric distribution is the following:

**2 - 0,2 mm = 92 %**
**0.2 - 0,1 mm = 5 %**
**0,1 - 0 mm = 3 %**

Analysis according to AFNOR of each of the granular classes by laboratory A has found:

- on 2 - 0.2 m -- Being given the larger of the grains, only a sulfo-hydrofluoric attack has been made and doubled

SiC = 98.00 - 98.97

Free $SiO_2$ = 0.90 - 0.75

- on 0.2 - 0.1 mm

| Sulfo-hydrofluoric attack | Oxidizing attack |
|---|---|
| SiC = 97.40 | $SiO_2$ = 142.00 |
| Free $SiO_2$ = 1.60 | SiC = $\frac{142.00 - 1.60}{1.5} = 93.70$ |

- on 0.1 - 0 mm

| Sulfo-hydrofluoric attack | Oxidizing attack /81 |
|---|---|
| SiC = 64.70 | Total $SiO_2$ = 111.20 |
| Free $SiO_2$ = 14.20 | SiC = $\frac{111.20 - 14.20}{1.5} = 64.70$ |

In calculating the average of SiC from these 3 analyses in relation to granulametric distribution of the sample, one finds:

$$\begin{array}{rcr} 0.92 \times 98.00 & = & 90.20 \\ 0.05 \times 97.40 & = & 4.87 \\ 0.03 \times 64.70 & = & \underline{1.94} \\ & & 97.01 \end{array}$$

3.2.3 For comparison, the FEPA method has been tried by laboratory A. One recalls that silicon carbide is not ground, but is screened; the sample taken of 5 g is reconsituted by weight according to the percentages of each granular class.

The sulfo-hydrofluoric attack (similar to that of AFNOR):

$SiC = 97.00$

Free $SiO_2 = 1.40$

This analysis cuts across the results found previously with the AFNOR method on screened silicon carbide.

3.2.4. For comparison, an analysis with X-ray diffraction has been made on a screened fraction 0.20-0.1 mm, which has been reground to 40 microns for the requirements of the test for laboratory B.

The content of SiC found is equal to 85%.

Grinding of the sample decreases the content of SiC which cuts across the results found in 3.1 by the AFNOR method on a sample ground of 125 microns.

### 3.3. Comparison of Results.

#### According to the AFNOR Method.

There is a difference in the contents of SiC between those attacked by sulfo-hydrofluoric and those attacked by an oxidizing agent; nevertheless, the results in the neighborhood of zero to four points make it possible to estimate an approximate objective value.

Analysis of a sample ground to 0.125 mm gives totally different results from those of a non-ground, but screened sample. In particular, one can note the much lower content of SiC in the 0.1-0 mm fraction in relation to the 0.2-0.1 mm fraction which is the next higher.

### Between the AFNOR and FEPA Method

There is a difference of 15 points in the titration of SiC and 10 points in titration of $SiO_2$.

The AFNOR and FEPA chemical attacks were similar to those demonstrated above, only the grinding of the sample is involved.

### 3.4. Conclusion.

Being given that the lower content of SiC determined by analysis following the AFNOR method does not seem more imputable than grinding of the same sample and cannot be explained by breakdown of the first material, without the causes being determined, it appears necessary to titrate the content of SiC and silicon carbide either by the FEPA method or by the AFNOR method on a non-ground sample but one which is screened and reconstituted granulometrically.

## 4. Analysis of Refractory Products Based on Silicon Carbide

Two refractory products R1 and R2 have been analysed according to the AFNOR and FEPA methods and by X-ray diffraction, by laboratories A and B.

### 4.1. AFNOR Method /82

The sulfo-hydrofluoric attack following recovery of the

SiC by oxidizing attack makes it possible to subtract the total impurities which remain from the sulfo-hydrofluoric attack.

| | Product R 1 | | Product R 2 |
|---|---|---|---|
| | Laboratory A | Laboratory B | Laboratory B |
| SiC | 71.80 | 73.60 | 54.70 |
| Free C | traces | - | - |
| Free $SiO_2$ | 11.20 | 10.00 | 31.00 |
| Free Si | - | 0.34 | 0.17 |
| $Al_2O_3$ | 15.80 | 14.60 | 12.30 |
| $TiO_2$ | 0.30 | 0.18 | 0.15 |
| $Fe_2O_3$ | 0.60 | 0.30 | 0.24 |
| CaO | 0.40 | 0.45 | 0.45 |
| MgO | 0.36 | 0.32 | 0.16 |
| $Na_2O$ | 0.14 | 0.21 | 0.23 |
| $K_2O$ | 0.10 | 0.12 | 0.14 |

In order to encompass in a more precise fashion the content of SiC, laboratory B has carried out counteranalyses according to the AFNOR method on the R1 and R2 products.

The results found by these new analyses for other compounds than those containing silicon (Column 1) are, in regard to preceding results, the following:

| | R 1 | | R 2 | |
|---|---|---|---|---|
| | I | II | I | II |
| $Al_2O_3$ | 14.62 | 14.60 | 12.15 | 12.30 |
| $TiO_2$ | 0.18 | 0.18 | 0.15 | 0.15 |
| $Fe_2O_3$ | 0.30 | 0.30 | 0.24 | 0.24 |
| CaO | 0.34 | 0.45 | 0.11 | 0.45 |
| MgO | 0.36 | 0.32 | 0.16 | 0.16 |
| $K_2O$ | 0.12 | 0.12 | 0.14 | 0.14 |
| $Na_2O$ | 0.21 | 0.21 | 0.23 | 0.23 |
| Total | 16.13 | 16.18 | 13.18 | 13.67 |

These values show a very good agreement, but the problem is complicated by the question of distribution of the silicon between them.

Laboratory B, after many counteranalyses with sulfo-hydrofluoric and oxidizing attacks, was not able to determine with certainty the precise values of SiC and $SiO_2$ contained in the R1 and R2 products. The disparities found are of the same order as those concerning technical silicon carbide in 3.1.

## 4.2. FEPA Method

On product R1 by laboratory A.

Being given the fact that this method does not involve silicon carbide and not the products of silicon carbide, titration has been carried out, out of curiosity, after succinic grinding of the sample of crushed grains in which the granulometric condition is the following:

70% of 3.15 - 0.630 mm
15% of 0.630 - 0.250 mm
15% of 0.250 - 0 mm

After reconstitution by weight of the sample and sulfo-hydrofluoric attack, the result obtained SiC = 81.87% is too high.

In effect, the residue of SiC contains impurities which it is suitable to titrate by oxidizing attack. We find:

| | | |
|---|---|---|
| Pure SiC | = | 73.72 |
| $Al_2O_3$, principally | = | 8.15 |
| | | 81.87 |

The titration of $Al_2O_3$ on the filtrate of the residue gives 6.47%, total aluminum will equal: 8.15 + 6.47 = 14.62.

In other words: free $SiO_2$ + free C = 11.44.

The analysis is then the following:

SiC = 73.72
Total $SiO_2$ + free C = 11.44
$Al_2O_3$, principally = 14.62

In spite of the grinding which has been done, the results essentially cut across those found by the AFNOR method.

## 4.3. X-ray Diffraction Method.

Analysis of the R1 and R2 products by laboratory B:

/83

| | R1 Product | R2 Product |
|---|---|---|
| Silicon Carbide | 63% | 44% |
| Mullite | 11% | 20% |
| Cristobalite | 9% | 24% |
| Tridymide | - | 5% |
| Corundum | 6% | 0.5% |
| Quartz | 2.5% | - |

A difference of 10 points has been noted between the contents of SiC determined by chemical analysis and by X-ray diffraction.

## 4.4. Comparison of Results

### According to the AFNOR and FEPA Methods

The contents of SiC determined are approximate and the condition for recovering the residue of SiC by oxidizing attack for subtracting impurities, is not indicated by the FEPA method because the analysis is not concerned with silicon carbide. Nevertheless, it has been shown that the effect of grinding does not interfere.

Analysis by X-ray diffraction decreases by 10 points the content of SiC in relation to chemical analysis.

### 4.5. Conclusion.

Given that the effect of grinding does not appear on the results of chemical analyses and that the content of SiC determined corresponds approximately to the quantity of technical silicon carbide introduced into the product, it appears necessary to titrate the content of SiC from a product with a base of silicon carbide by the AFNOR method.

## 5. Conclusions and Summary

The experimental results concern titration of pure silicon carbide (SiC) in technical silicon carbide and in products based on technical silicon carbide and show that differentiation must be made between the methods considered.

In the case of technical silicon carbide, the discrepancies stated follow the methods used, AFNOR and FEPA chemical analyses and analysis by X-ray diffraction; these seemed caused by grinding of the sample. The causes of this effect have not been studied given the essentially practical purpose of this work.

In the case of products based on technical silicon carbide, the methods of chemical analysis give somewhat closer results but the analysis by X-ray diffraction seems too imprecise for quantitative titration.

The crossover of results in determination of content of pure SiC from technical silicon carbide and determination of the content of pure SiC in products containing a known quantity of technical silicon carbide has made it possible to choose among the proposed methods of analyses.

The best agreement is obtained by use of FEPA chemical analysis for technical silicon carbide and AFNOR chemical analysis for products based on technical silicon carbide.