U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE 112

SILICON CARBIDE

(All results are based on a sample dried at 105 to 110° C.)

Analyst [*]	Total silicon	Total carbon	Free carbon	Silicon carbide	Fe	Al	Ti	Zr	Ca	Mg
1	69. 09	29. 15	0. 09	97. 01	0. 44	0. 24	0. 024	0. 025	<0. 01	0. 02
2	69. 12	29. 04	. 08	{ 96. 68 / 97. 03[a] }	. 46	. 35[b]			. 04	. 02
3	69. 10	29. 07	. 10	96. 71	. 45	. 22	. 024	. 027	. 02	. 02
4	69. 12	29. 08	. 10	96. 75	. 45	. 23	. 027	. 028	. 04	. 02
5	69. 10	29. 14	. 10	96. 94	. 46	. 244[c]	. 026		. 04	. 03
General average [d]	69. 11	29. 10	. 09	96. 85	. 45	. 23	. 025	. 027	. 03	. 02

[a] Value obtained by treating a sample with $HF—HNO_3—H_2SO_4$ to remove free SiO_2, igniting, fusing for 10 minutes with $K_2S_2O_7$, dissolving the melt in hot dilute HCl, filtering, washing, and igniting the residue at 750 to 850° C. Includes correction for any SiC decomposed during fusion.

[b] Includes Ti and Zr and is omitted from general average.

[c] Includes Zr and is omitted from general average.

[d] All results were calculated by using the International Atomic Weights of 1935. Since then, the atomic weight for carbon has been changed from 12.00 to 12.01. Corrected to this basis the values for total carbon and silicon carbide become 29.12 and 96.86, respectively.

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Director.

METHODS USED AT THE NATIONAL BUREAU OF STANDARDS IN THE ANALYSIS OF STANDARD SILICON CARBIDE 112

Total Silicon.—Mix 0.5 g of silicon carbide, dried at 105° C, with 3.5 g of potassium nitrate and 20 g of sodium carbonate in a large platinum crucible which has been lined with 2 g of sodium carbonate. Cover the crucible and fuse slowly. Dissolve the melt in water, add an excess of dilute sulphuric acid (1+1), evaporate to fumes of sulphuric acid and determine silica in the customary manner, including any recovery from the R_2O_3 precipitate. Calculate the percentage of silicon using the factor 0.4672.

Total Carbon.—Transfer an accurately weighed sample of approximately 0.25 g of dry silicon carbide to a small, glass stoppered, cylindrical weighing bottle. Add 2 g of an accelerator, such as Pb_3O_4 (C and CO_2 free) or powdered metallic copper, and thoroughly mix by gentle shaking. Fill a combustion boat (approximately 5 inches by ¾ inch) with RR Alundum (90 mesh) or Alfrax 15A (80 to 150 mesh) and by means of a ⅜-inch glass rod or other suitable tool, groove or furrow the bedding. Line thinly the groove with 0.5 g of the accelerator. Place the mixed sample, thinly and evenly, in the boat thus prepared, rinse the weighing bottle with a few small portions of Alundum or Alfrax and cover the entire charge with a thin layer of the same. Place the boat and contents in a combustion furnace at 1,050 to 1,100° C, preheat for 1 minute, admit oxygen as in the determination of carbon in steel and burn for 45 minutes. Absorb the carbon dioxide in Ascarite, cool and weigh. Correct the result thus found by blank determinations for the furnace, oxygen, boat, bedding material, and accelerator. Calculate the percentage of carbon by use of the factor 0.2729.

Free Carbon.—Transfer 4 g of dried silicon carbide to an empty combustion boat, ignite in oxygen at a temperature of 900 to 915° C for 15 minutes and absorb the carbon dioxide in Ascarite. Cool and weigh. Correct the result thus found by blank determinations for the furnace, oxygen, and boat, and calculate the percentage of carbon by use of the factor 0.2729.

Silicon Carbide.—Deduct the value obtained for free carbon from the value found for total carbon. Calculate the remainder to silicon carbide, using the factor 3.3364.

Iron.—Fuse 2 g of the dried carbide with a mixture of 20 g of sodium carbonate and 15 g of potassium nitrate as under the determination of silicon. Dissolve the melt in water and if an unattacked residue is observed, filter, ignite, and again fuse with sodium carbonate and potassium nitrate. Remove the silica by evaporating with an excess of sulphuric acid in the customary manner and recover any nonvolatile residue that remains after volatilizing the silica with hydrofluoric and sulphuric acids. Treat the filtrate from the silica separation with hydrogen sulphide and filter to remove platinum and other insoluble sulphides. Boil the filtrate to expel hydrogen sulphide, oxidize with bromine water and precipitate the iron, aluminum, titanium, and zirconium twice with ammonium hydroxide. Reserve the filtrate for the determination of calcium and magnesium. Ignite and weigh the mixed oxides after treatment with hydrofluoric and sulphuric acids to remove silica. Calculate any silica thus found to silicon and add the amount to that found under total silicon. Fuse the mixed oxides with potassium pyrosulphate, dissolve the melt in dilute sulphuric acid, and precipitate the iron as sulphide from an ammoniacal ammonium tartrate solution. Filter, wash, and reserve the filtrate for the determination of titanium and zirconium. Ignite the precipitate and calculate iron using the factor 0.6994.

Titanium.—Add sulphuric acid to the reserved filtrate until present in 10 percent by volume. Precipitate with cupferron. Decompose the precipitate with sulphuric and nitric acids, and heat the solution until all organic matter is destroyed and nitric acid is expelled. Determine the titanium by adding hydrogen peroxide and comparing the color with that of a known standard and reserve the solution.

Zirconium.—Precipitate the zirconium as phosphate in the solution used for the determination of titanium Filter and wash with a 5-percent solution of ammonium nitrate. Ignite the precipitate and calculate the Zr, using the factor 0.3439.

Aluminum.—Subtract the combined weight of iron, titanium, and zirconium, calculated as oxides, from the weight of the combined oxides as determined under iron. Calculate the remainder as aluminum, using the factor 0.5291.

Calcium and Magnesium.—Concentrate the combined filtrate obtained in the ammonium hydroxide separations and precipitate the calcium and magnesium twice as phosphates. Ignite the combined phosphates and weigh. Dissolve the phosphates in a little dilute sulphuric acid and precipitate the calcium as sulphate by the addition of 75 percent ethyl alcohol. Filter, wash with 75 percent ethyl alcohol containing a few drops of sulphuric acid, dissolve the precipitate in a very little dilute hydrochloric acid, precipitate as oxalate in as small a volume as possible, and ignite to oxide. Calculate the percentage of calcium by use of the factor 0.7147. Subtract from the weight of the combined phosphates the tricalcium phosphate equivalent of the calcium found. Calculate the remainder as magnesium using the factor 0.2184.

Attempts were made to determine other forms in which silicon might be present, such as free silicon, silica, and silicon monoxide, but owing to the uncertainty of the analytical methods involved, reliable values for these constituents were not obtained.