

**The Technical Association of Refractories, Japan**  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 1(Silicon Carbide, SiC 100)**  
**Results of Analyses**

Unit:mass%

Component	Certified value		Approximate value								
	Total Carbon	Free Carbon	Residue after HF treatment	Aluminum	Iron	Titanium	Calcium	Magnesium	Free Silicon	Oxygen	Nitrogen
Chemical Symbol	C <sub>total</sub>	C <sub>free</sub>	SiC <sub>HF</sub>	Al	Fe	Ti	Ca	Mg	Si <sub>free</sub>	O	N
Certified and Aproximate	29,81	0,04	99,58	0,008	0,044	0,0035	<0,001	<0,001	0,06	0,048	0,030
Laboratory L <sub>1</sub>	29,78	.....	99,58	0,0069	0,0418	0,0032	0,0004	0,0002	0,038	0,0457	0,024
L <sub>2</sub>	29,78	0,043	.....	0,0068	0,0438	0,0032	0,0000	0,0000	0,067	.....	.....
L <sub>3</sub>	29,82	0,041	99,45	.....	.....	.....	.....	.....	.....	.....	.....
L <sub>4</sub>	29,88	0,036	99,60	.....	.....	.....	.....	.....	.....	.....	.....
L <sub>5</sub>	29,75	0,030	99,58	0,0096	0,0464	0,0036	0,0004	0,0004	0,051	.....	.....
L <sub>6</sub>	29,80	.....	99,66	.....	.....	.....	.....	.....	.....	.....	.....
L <sub>7</sub>	29,82	0,039	99,65	0,0090	0,0482	0,0042	0,0001	0,0001	0,090	0,0575	0,036
L <sub>8</sub>	29,74	.....	99,50	.....	.....	.....	.....	.....	0,080	.....	.....
L <sub>9</sub>	29,78	0,036	.....	.....	.....	.....	.....	.....	.....	.....	.....
L <sub>10</sub>	29,80	0,054	99,62	0,0076	0,0424	0,0033	<0,001	<0,0005	.....	.....	.....
L <sub>11</sub>	29,92	0,04	.....	.....	.....	.....	.....	.....	.....	0,0502	0,0294
L <sub>12</sub>	.....	.....	.....	.....	.....	.....	.....	.....	.....	0,0454	.....
L <sub>13</sub>	.....	.....	.....	.....	.....	.....	.....	.....	.....	0,0436	.....
Average ( $\bar{X}$ )	29,806	0,0399	99,58	0,008	0,0445	0,0035	.....	.....	0,065	0,0485	0,0298
Standard deviation											
Reproducibility $s_{\bar{x}}$	0,055	0,0072	0,072	0,0013	0,0027	0,0004	.....	.....	0,025	0,0058	.....
Reproducibility $s_{(I)}$ within laboratory	0,040	0,0032	0,044	0,0002	0,0034	0,0002	.....	.....	0,003	0,0006	.....
Uncertainty C(95%) <sup>a2</sup>	0,04	0,01	0,06	0,002	0,003	0,0005	.....	.....	0,03	0,007	.....

(Note) \*1  $s_{(I)}$  is time-differenet intermediate precision standard deviation.\*2 The half-width confidence interval  $C(95\%) = t_{I-1,005} \times s_{\bar{x}} / \sqrt{I}$  ( $I$  : number of laboratories)

(1) Laboratories ; Krosaki Corporation(now, Kuroasaki Harima Corporation), Kawasaki Refractories Co., Ltd.,

Nihon Tokushu Rozai Co., Ltd., Taiko Refractories Co., Ltd., Asahi Glass Co., Ltd., Harima Ceramic Co., Ltd.

(now, Kuroasaki Harima Corporation), Shinagawa Refractories Co., Ltd., Toshiba Ceramics Co., Ltd.,

TYK Corporation, Showa Denko K.K., Horiba, Ltd., Japan Analyst Corporation, Shimadzu Corporation

(2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))

① F.C : determined by method using mass gain correction owing to oxidation of SiC.

② SiC<sub>HF</sub> : filtrate residue which is ignited at 780~800°C for 40 min after HF(H<sub>2</sub>SO<sub>4</sub>) treatment.③ Metal element except Al were decomposed HF+H<sub>2</sub>SO<sub>4</sub>) decompisition in pressure vessel and determined by ICP-AES and AAS. Al was fused with sodium carbonate, treated by HF+H<sub>2</sub>SO<sub>4</sub> and determined by ICP-AES.

(3) Each analytical value is the average of two values obtained by two measurements on different days.

(4) Outlier tests were carried out by Grubbs test method. The samples rejected by Grubbs tests were discussed in view of analytical techniques and it was decided whether they should be adopted or not.

(5) Date of preparation : December, 1993

Prepared, Values  
given and Certified by**The Technical Association of Refractories, Japan**

New Ginza Bldg., 3-13, Ginza 7-chome, Chuo-ku, Tokyo 104-0061, Japan

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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 2(Carbon Containing Material, C 5)**  
**Results of Analyses**

Unit : mass%

Component	Certified value		Approximate value
	Total Carbon	Free Carbon	
Chemical Symbol	T.C	F.C	LOI
Certified and Aproximate	5,03	4,98	5,11
Laboratory L <sub>1</sub>	5,025	4,974	5,169
L <sub>2</sub>	5,078	5,028	5,081
L <sub>3</sub>	5,018	5,040	.....
L <sub>4</sub>	4,997	4,944	5,086
L <sub>5</sub>	4,964	4,928	5,164
L <sub>6</sub>	5,040	4,985	5,112
L <sub>7</sub>	5,023	4,897	5,058
L <sub>8</sub>	5,040	5,020	.....
L <sub>9</sub>	5,106	4,997	5,088
Average $\bar{X}$	5,032 3	4,979 2	5,108 3
Standard deviation			
Reproducibility $s_{\bar{X}}$	0,041 8	0,048 3	0,042 8
Reproducibility $s_{(T)}^{*1}$ within laboratory	0,022 9	0,033 1	0,029 7
Uncertainty C(95%) <sup>*2</sup>	0, 04	0,04	0,04

(Note) \*1  $s_{(T)}$  is time-differenet intermediate precision standard deviation.

\*2 The half-width confidence interval  $C(95\%) = t_{l-1,0.05} \times s_{\bar{X}} / \sqrt{l}$  (l : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefractories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
- (2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))
- (3) Each analytical value is the average of two values obtained by two measurements on different days.
- (4) Outlier tests were carried out by Grubbs test method. The samples rejected by Grubbs tests were discussed in view of analytical techniques and it was decided whether they should be adopted or not.
- (5) Date of preparation :November, 2004

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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 3(Carbon Containing Material, C 10)**  
**Results of Analyses**

Unit:mass%

Component	Certified value		Approximate value
	Total Carbon	Free Carbon	
Chemical Symbol	T.C	F.C	LOI
Certified and Aproximate	10,06	10,01	10,11
Laboratory L <sub>1</sub>	10,02	9,980	10,21
L <sub>2</sub>	10,10	9,954	10,08
L <sub>3</sub>	10,05	10,09	.....
L <sub>4</sub>	10,05	9,980	10,14
L <sub>5</sub>	10,06	9,985	10,12
L <sub>6</sub>	10,12	10,07	10,07
L <sub>7</sub>	10,02	10,03	10,09
L <sub>8</sub>	10,02	10,10	.....
L <sub>9</sub>	10,08	9,922	10,03
Average $\bar{X}$	10,058	10,012	10,106
Standard deviation			
Reproducibility $s_{\bar{x}}$	0,036 9	0,063 8	0,057 8
Reproducibility $s_{(r)}$ <sup>*1</sup> within laboratory	0,032 7	0,036 1	0,015 4
Uncertainty C(95%) <sup>*2</sup>	0,03	0,05	0,06

(Note) \*1  $s_{(r)}$  is time-differenet intermediate precision standard deviation.

\*2 The half-width confidence interval  $C(95\%) = t_{l-1,0.05} \times s_{\bar{x}} / \sqrt{l}$  (l : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefractories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
- (2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))
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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 4(Carbon Containing Material, C 20)**  
**Results of Analyses**

Unit : mass%

		Certified value		Approximate value
Component		Total Carbon	Free Carbon	Loss on Ignition
Chemical Symbol		T.C	F.C	LOI
Certified and Aproximate		20,04	19,92	20,01
Laboratory	L <sub>1</sub>	19,98	19,83	20,02
	L <sub>2</sub>	19,94	19,90	20,04
	L <sub>3</sub>	19,98	20,02	.....
	L <sub>4</sub>	20,04	19,91	20,06
	L <sub>5</sub>	19,98	19,76	20,02
	L <sub>6</sub>	20,08	19,97	19,98
	L <sub>7</sub>	20,12	20,08	20,04
	L <sub>8</sub>	20,18	19,94	.....
	L <sub>9</sub>	20,04	19,84	19,92
Average	$\bar{X}$	20,038	19,917	20,011
Standard deviation				
Reproducibility	$s_x$	0,080	0,100	0,051
Reproducibility within laboratory	$s_{I(T)}^{*1}$	0,045	0,094	0,033
Uncertainty	$C(95\%)^{*2}$	0,07	0,08	0,05

(Note) \*1  $s_{I(T)}$  is time-differenet intermediate precision standard deviation.

\*2 The half-width confidence interval  $C(95\%) = t_{I-1,0.05} \times s_{\bar{X}} / \sqrt{I}$  (I : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefractories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
- (2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))
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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 5(Carbon Containing Material, C 30)**  
**Results of Analyses**

Unit:mass%

		Certified value		Approximate value
Component		Total Carbon	Free Carbon	Loss on Ignition
Chemical Symbol		T.C	F.C	LOI
Certified and Aproximate		29,93	29,81	29,95
Laboratory	L <sub>1</sub>	29,88	29,70	30,06
	L <sub>2</sub>	29,97	29,74	29,96
	L <sub>3</sub>	29,87	29,89	.....
	L <sub>4</sub>	29,92	29,72	29,96
	L <sub>5</sub>	30,02	29,73	30,04
	L <sub>6</sub>	29,84	30,03	29,84
	L <sub>7</sub>	29,94	29,82	29,91
	L <sub>8</sub>	29,94	29,81	.....
	L <sub>9</sub>	30,01	29,88	29,90
Average	$\bar{X}$	29,932	29,813	29,953
Standard deviation				
Reproducibility	$s_{\bar{X}}$	0,062	0,107	0,077
Reproducibility within laboratory	$s_{t(r)}$ <sup>*1</sup>	0,088	0,145	0,086
Uncertainty	$C(95\%)^{*2}$	0,05	0,09	0,08

(Note) \*1  $s_{t(r)}$  is time-differenet intermediate precision standard deviation.\*2 The half-width confidence interval  $C(95\%) = t_{l=1,0.05} \times s_{\bar{X}} / \sqrt{l}$  (l : number of laboratories)

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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 6(Carbon Containing Material, C 50)**  
**Results of Analyses**

Unit:mass%

		Certified value		Approximate value
Component		Total Carbon	Free Carbon	Loss on Ignition
Chemical Symbol		T.C	F.C	LOI
Certified and Aproximate		49,99	49,97	49,95
Laboratory	L <sub>1</sub>	49,86	49,95	50,17
	L <sub>2</sub>	50,07	49,91	50,04
	L <sub>3</sub>	50,10	49,97	.....
	L <sub>4</sub>	50,19	49,93	50,02
	L <sub>5</sub>	49,96	50,00	49,97
	L <sub>6</sub>	49,86	49,93	49,96
	L <sub>7</sub>	49,93	50,12	49,92
	L <sub>8</sub>	50,20	50,01	.....
	L <sub>9</sub>	49,76	49,88	49,59
Average	$\bar{X}$	49,992	49,967	49,953
Standard deviation				
Reproducibility $s_x$		0,155	0,069	0,179
Reproducibility $s_{(t)}^{*1}$ within laboratory		0,144	0,192	0,105
Uncertainty	$C(95\%)^{*2}$	0,12	0,06	0,08

(Note) \*1  $s_{(t)}$  is time-differenet intermediate precision standard deviation.

\*2 The half-width confidence interval  $C(95\%) = t_{l-1,0.05} \times s_{\bar{X}} / \sqrt{l}$  (l : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefraactories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
- (2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))
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## The Technical Association of Refractories, Japan

**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 7(Carbon Containing Material, C 10-SiC 90)**  
**Results of Analyses**

Unit:mass%

		Certified value		Approximate value
Component		Total Carbon	Free Carbon	Silicon Carbide
Chemical Symbol		T.C	F.C	SiC
Certified and Aproximate		36,75	10,01	89,29
Laboratory	L <sub>1</sub>	36,72	9,988	89,26
	L <sub>2</sub>	36,74	10,032	89,18
	L <sub>3</sub>	36,78	9,966	89,52
	L <sub>4</sub>	36,77	10,002	89,36
	L <sub>5</sub>	36,84	9,930	89,82
	L <sub>6</sub>	36,74	10,042	89,14
	L <sub>7</sub>	36,70	10,055	88,95
	L <sub>8</sub>	36,81	10,160	88,97
	L <sub>9</sub>	36,66	9,889	89,37
Average	$\bar{X}$	36,751	10,007	89,286
Standard deviation				
Reproducibility $s_x$		0,054	0,079	0,273
Reproducibility $s_{I(r)}$ <sup>*1</sup> within laboratory		0,102	0,032	0,347
Uncertainty	$C(95\%)^{*2}$	0,05	0,07	0,22

(Note) \*1  $s_{I(r)}$  is time-differenet intermediate precision standard deviation.\*2 The half-width confidence interval  $C(95\%) = t_{l-1,0.05} \times s_{\bar{x}} / \sqrt{l}$  (l : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefractories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
- (2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))  
Silicon Carbide values are calculated by the following equation; SiC=3.3383 · (T.C-F.C)
- (3) Each analytical value is the average of two values obtained by two measurements on different days.
- (4) Outlier tests were carried out by Grubbs test method. The samples rejected by Grubbs tests were discussed in view of analytical techniques and it was decided whether they should be adopted or not.
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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 8 (Carbon Containing Material, C 5-SiC 30)**  
**Results of Analyses**

Unit: mass%

		Certified value		Approximate value
Component		Total Carbon	Free Carbon	Silicon Carbide
Chemical Symbol		T.C	F.C	SiC
Certified and Aproximate		14,12	5,21	29,74
Laboratory	L <sub>1</sub>	14,17	5,286	29,66
	L <sub>2</sub>	14,09	5,264	29,46
	L <sub>3</sub>	14,12	5,264	29,54
	L <sub>4</sub>	14,13	5,161	29,96
	L <sub>5</sub>	14,07	5,152	29,77
	L <sub>6</sub>	14,09	5,250	29,48
	L <sub>7</sub>	14,08	5,106	29,98
	L <sub>8</sub>	14,16	5,244	29,77
	L <sub>9</sub>	14,17	5,174	30,04
Average	$\bar{X}$	14,12	5,211 2	29,74
Standard deviation				
Reproducibility	$s_{\bar{X}}$	0,039	0,063 5	0,218
Reproducibility within laboratory	$s_{t(r)}^{*1}$	0,032	0,016 8	0,133
Uncertainty	$C(95\%)^{*2}$	0,03	0,05	0,17

(Note) \*1  $s_{t(r)}$  is time-differenet intermediate precision standard deviation.

\*2 The half-width confidence interval  $C(95\%) = t_{l-0.05} \times s_{\bar{X}} / \sqrt{l}$  (l : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefractories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
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The Technical Association of Refractories, Japan  
**Certified Reference Material for Carbon Analysis of C/SiC Refractories**  
**J R R M 1 0 0 9(C/SiC Containing Material, C 40-SiC6)**  
**Results of Analyses**

Unit : mass%			
Component	Certified value		Silicon Carbide
	Total Carbon	Free Carbon	
Chemical Symbol	T.C	F.C	SiC
Certified and Aproximate	39,43	37,67	6,18
Laboratory L <sub>1</sub>	39,36	37,54	6,176
L <sub>2</sub>	39,40	37,92	6,219
L <sub>3</sub>	39,30	37,61	6,238
L <sub>4</sub>	39,42	37,62	6,171
L <sub>5</sub>	39,15	37,62	6,052
L <sub>6</sub>	39,56	37,83	6,091
L <sub>7</sub>	39,53	37,71	6,076
L <sub>8</sub>	39,29	37,82	.....
L <sub>9</sub>	39,84	37,33	6,412
Average $\bar{X}$	39,428	37,667	6,179 4
Standard deviation			
Reproducibility $s_{\bar{X}}$	0,201	0,178	0,116
Reproducibility within laboratory $s_{(t)}^{*1}$	0,115	0,152	0,054
Uncertainty C(95%) <sup>*2</sup>	0,16	0,14	0,09

(Note) \*1  $s_{(t)}$  is time-differenet intermediate precision standard deviation.

\*2 The half-width confidence interval  $C(95\%) = t_{1-0.05} \times s_{\bar{X}} / \sqrt{l}$  (l : number of laboratories)

- (1) List of laboratories: Krosaki Harima Corporation, JFE Refractories Corporation, TaikoRefractories Co., Ltd., Asahi Glass Ceramics Co., Ltd., Shinagawa Refractoties Co., Ltd., TYK Corporation, Okayama Ceramics Research Foundation, Horiba, Ltd., LECO Japan Corporation
- (2) Analytical methods ; JIS R 2011(Methods for chemical analysis of refractories containing carbon and/or silicon-carbide))  
 The C values for Silicon Carbide values were determinated from the remain sample for F.C determination, and were calculated by the following equation; SiC=3.3383•T.C
- (3) Each analytical value is the average of two values obtained by two measurements on different days.
- (4) Outlier tests were carried out by Grubbs test method. The samples rejected by Grubbs tests were discussed in view of analytical techniques and it was decided whether they should be adopted or not.
- (5) Date of preparation :November, 2004

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