

UNITED STATES DEPARTMENT OF COMMERCE
WASHINGTON 25, D.C.

National Bureau of Standards
Certificate of Analyses
of
Standard Sample No. 103 a
Chrome Refractory

(All results are based on a sample dried for 2 hours at 105 to 110° C)

Analyst	Cr ₂ O ₃	Al ₂ O ₃	Total iron as FeO	MnO	MgO	CaO	SiO ₂	TiO ₂	ZrO ₂	P ₂ O ₅
1.....	^a 32.05	^b 29.95	^c 12.43	^d 0.12	^e 18.57	^f 0.70	^g 4.68	^h 0.22	ⁱ 0.01	^j 0.007
2.....	^k 31.95	^l 30.10 ^m 30.01	ⁿ 12.43	.12	18.50	.69	^o 4.68	.2101
3.....	^p 32.07 ^q 32.09	^r 30.02 ^s 29.95	^t 12.45	^u .11	18.51	^v .68 ^w .67	^x 4.59	^y .22	^z <.004
4.....	^d 32.08	^g 30.05	^{a'} .10	18.62	^{b'} .60	^{c'} 4.58	^{e'} .20	^{d'} .017
5.....	^{e'} 31.98	^c 12.43	^g 4.63
6.....	^d 32.10	^{t'} 29.91	^{h'} 12.40	^{i'} .09	^e 18.45	^f .72	^o 4.64	^{i'} .21	ⁱ .01	^{j'} .01
7.....	32.12	29.8510	18.49	.7025
8.....	^p , ^{r'} 31.78	^{k'} 29.80	^{l'} 12.43	^{m'} .10	^{n'} 18.63	^{b'} .74	^o 4.60	^{o'} .23	^{p'} .009 ^{q'} .009
Average.....	32.06	29.96	12.43	0.11	18.54	0.69	4.63	0.22	0.01	0.01

^a Persulfate oxidation and potentiometric titration with ferrous ammonium sulfate. Corrected for vanadium.
^b Dissolved in perchloric acid, volatilized chromium with hydrochloric acid, extracted R₂O₃ cupferrates with chloroform at pH 3.5, separated aluminum by extracting R₂O₃ cupferrates in 4N hydrochloric acid with chloroform, precipitated aluminum with ammonia and weighed as Al₂O₃.
^c Dissolved in perchloric acid. SnCl₂-K₂Cr₂O₇.
^d Dissolved in perchloric acid. Periodate colorimetric method.
^e Dissolved in perchloric acid. Gravimetric phosphate method. Corrected for Ca and Mn.
^f Precipitated with magnesium, separated as sulfate in alcohol, precipitated with oxalate and weighed as CaO.
^g Perchloric acid dehydration.
^h Peroxide colorimetric method. Used 2d chloroform extraction footnote "b."
ⁱ Used R₂O₃ precipitate, separated with cupferron, and determined gravimetrically as phosphate.
^j Dissolved in perchloric acid, precipitated with ammonia and determined by molybdenum blue colorimetric method.
^k Average of footnote "a" and leached sodium peroxide fusion with water, filtered, acidified filtrate, added excess ferrous ammonium sulfate and titrated with permanganate.
^l Precipitated with phenylhydrazine. Corrected for Cr₂O₃, Fe₂O₃, TiO₂, and P₂O₅.
^m Separated with cupferron, corrected for Cr₂O₃ and P₂O₅.
ⁿ Leach sodium peroxide fusion, filtered and dissolved precipitate. SnCl₂-KMnO₄.
^o Perchloric acid-sulfuric acid dehydration.
^p Reduced with excess ferrous iron and titrated with permanganate.
^q Reduced with excess ferrous iron and titrated with dichromate. Corrected for vanadium.
^r Separated with ammonia and mercury cathode and precipitated with ammonia. Corrected for TiO₂.

^{a'} Removed chromium by ion exchange, separated with ammonia and mercury cathode and precipitated with ammonia. Corrected for TiO₂.
^{b'} Leached sodium peroxide-sodium hydroxide fusion, reprecipitated with ammonia. SnCl₂-K₂Cr₂O₇.
^{c'} Leached sodium peroxide fusion and dissolved hydroxides. Periodate colorimetric method.
^{d'} Used filtrate of ammonia precipitation (footnote "i"), precipitated as phosphate, separated as sulfate in alcohol and determined as oxalate.
^{e'} Fused with sodium peroxide and dehydrated with perchloric acid.
^{f'} Fused with sodium peroxide. Ammonia and cupferron separations made. Peroxide colorimetric method.
^{g'} Fused with sodium peroxide and reduced chromium with alcohol. Phosphomolybdate volumetric method.
^{h'} Leached sodium peroxide fusion, precipitated with NH₄Cl, reprecipitated with ammonia, dehydrated with perchloric acid, filtered, and precipitated with ammonia.
^{i'} Dissolved in perchloric acid and volatilized chromium with hydrochloric acid. Volumetric bismuthate method.
^{j'} Volumetrically. Oxalate titrated with permanganate.
^{k'} Dissolved in perchloric acid and made ammonia separation. Peroxide colorimetric method.
^{l'} Dissolved in perchloric acid, precipitated with ammonium molybdate, reduced with Jones reductor and titrated with permanganate.
^{m'} Oxidized with permanganate and titrated potentiometrically with ferrous ammonium sulfate. Corrected for vanadium.
^{n'} SiO₂ removed, R₂O₃ precipitated with NH₄OH. Acid cupferron separation, aluminum precipitated in filtrate with cupferron after neutralization with NH₄OH. Corrected for Cr₂O₃.
^{o'} Dissolved in perchloric and sulfuric acids, precipitated as sulfide, ignited, fused with potassium bisulfate, oxidized with permanganate and titrated with titanous chloride.

^{p'} Dissolved in perchloric and sulfuric acids, precipitated with magnesium and calcium, removed calcium with alcoholic sulfate separation. Bismuthate method.
^{q'} Used acid cupferrates of aluminum determination (footnote f') and made an ammonical tartrate, hydrogen sulfide separation. Peroxide colorimetric method.
^{r'} Gravimetric phosphomolybdate method.
^{s'} Fused with sodium carbonate-sodium borate and made a mercury cathode separation. Gravimetric 8-hydroxyquinoline method. Corrected for titanium.
^{t'} Dissolved in perchloric and sulfuric acids, precipitated with ammonia, and dissolved in hydrochloric acid. SnCl₂-K₂Cr₂O₇.
^{u'} In filtrate from silicon determination reduced chromium with sulfurous acid, reoxidized iron with nitric acid, precipitated with ammonia, made sodium carbonate separation and determined photometrically with periodate.
^{v'} Gravimetric as phosphate. Used filtrate from CaO determination.
^{w'} Prepared solution as in footnote "m." Peroxide colorimetric method.
^{x'} Dissolved in nitric, hydrochloric and perchloric acids, filtered, volatilized arsenic as bromide, and precipitated phosphorus as phosphomolybdate. Determined the equivalent molybdenum as lead molybdate.
^{y'} Dissolved in perchloric acid, volatilized arsenic as bromide and determine phosphorus colorimetrically as phospho-vanado-molybdate in iso-amyl alcohol.
^{z'} Omitted from average.
Analyst 1 and analyst 3 reported 0.09 percent vanadium and analyst 6 reported 0.12 percent V₂O₅.
Combustion in a tube furnace at approximately 900° C indicates 0.99 percent of water which is not removed by drying at 105 to 110° C.

List of Analysts

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| 1. Keith M. Sappenfield, National Bureau of Standards. | 6. B. C. Ruprecht and R. P. Lucas, Harbison-Walker Refractories Co., Pittsburgh, Pa. |
| 2. Paul J. Byler, Booth, Garrett & Blair, Philadelphia, Pa. | |
| 3. Andrew S. McCreath & Sons, Inc., Harrisburg, Pa. | |
| 4. John H. Montague, E. G. Lavino and Co., Norristown, Pa. | 7. L. J. Trostel, General Refractories Co., Baltimore, Md. |
| 5. George Oplinger, K. A. Lane, and M. S. Budd, Solvay Process Division, Allied Chemical Corp., Syracuse, N.Y. | 8. C. E. A. Shanahan, Richard Thomas & Baldwins, Ltd., Whitchurch, Aylesbury, Bucks, England. |

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A. V. ASTIN, Director.