U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses of

STANDARD SAMPLE 132

MOLYBDENUM–TUNGSTEN–CHROMIUM–VANADIUM STEEL

ANALYST*	C	Mn	P	S	Si	COPPER H_2S–CuS–CuO	NICKEL Weighed as nickel dimethylglyoxime	Cr	V	Mo		W
	Direct combustion	Persulfate-Arsenite	Alkali-Molybdate [a]	Gravimetric (direct oxidation and final precipitation in reduced solution)	Nitric-sulfuric acid dehydration			$FeSO_4$–$KMnO_4$ titration	HNO_3 oxidation, potentiometric titration in presence of W	Gravimetric	Colorimetric	
1	0. 798	[b] 0. 247	[c] 0. 028	0. 002	[d] 0. 232	[e] 0. 149	0. 095	[f] 4. 13	1. 63	[g] 7. 07	7. 11	[h] 6. 28
2	. 805	[i]. 24	[j]. 031	. 007	[k]. 225	----------	[l]. 094	4. 08	[m] 1. 66	[n] 7. 14	----------	6. 29
3	. 809	[op]. 236	. 024	. 005	[d]. 238	[q]. 154	. 085	[f] 4. 11	1. 61	[n] 7. 08	----------	[r] 6. 33
4	. 802	[ip]. 25	[jp]. 025	. 003	[kd]. 24	[qp]. 151	[sp]. 095	[t] 4. 11	[m] 1. 68	----------	7. 13	[h] 6. 36
5	. 809	. 26	. 029	. 007	. 238	. 15	. 098	4. 08	1. 62	7. 06	----------	6. 20
6	. 810	[up]. 251	. 025	[v]. 004	[d]. 243	[e]. 148	[l]. 096	4. 14	[m] 1. 64	[g] 7. 04	----------	[h] 6. 34
7	. 800	[ip]. 258	. 027	[v]. 006	[k]. 244	[e]. 160	. 098	4. 12	1. 61	[w] 7. 10	----------	[r] 6. 25
8	. 801	[x]. 26	[j]. 025	. 005	[y]. 235	[q]. 14	[s]. 09	[t] 4. 06	[z] 1. 60	[n] 7. 02	----------	6. 28
9	. 798	[i]. 252	. 026	. 004	. 242	[e]. 154	[s]. 092	[t] 4. 12	[m] 1. 67	[n] 7. 06	----------	[h] 6. 23
10	. 798	----------	----------	----------	[y]. 228	----------	----------	[f] 4. 11	1. 67	[z1] 7. 01	7. 04	[z1] 6. 34
----------	. 798	. 27	[z2]. 030	. 002	[y]. 26	[z3]. 136	[s]. 095	4. 10	[m] 1. 62	[n] 7. 09	----------	[h] 6. 27
Averages	**0. 803**	**0. 252**	**0. 027**	**0. 004**	**0. 239**	**0. 149**	**0. 094**	**4. 11**	**1. 64**	**7. 07**	**7. 09**	**6. 29**
General average	**0. 803**	**0. 252**	**0. 027**	**0. 004**	**0. 239**	**0. 149**	**0. 094**	**4. 11**	**1. 64**	**7. 07**		**6. 29**

[a] Tungsten removed and vanadium reduced. Phosphorus precipitated at 10° to 20° C, washed with a 1-percent solution of KNO_3, and titrated with alkali standardized by the use of acid potassium phthalate and the ratio 23 NaOH:1 P.

[b] Bismuthate ($FeSO_4$–$KMnO_4$) method after ZnO separation.

[c] Tungsten removed after HCl-HNO_3 digestion followed by fuming with $HClO_4$. Phosphorus precipitated with molybdate in hot nitric acid solution and ultimately weighed as $Mg_2P_2O_7$.

[d] Double dehydration.

[e] Finished by electrolysis.

[f] Persulfate oxidation, potentiometric titration with ferrous ammonium sulfate solution standardized with recrystallized potassium dichromate.

[g] α-Benzoinoxime method after removal of tungsten by acid digestion. Corrected for molybdenum occluded in tungsten, and the main molybdenum precipitate corrected for ammonia insoluble, and tungsten. See BS J. Research **9**, 1 (1932) RP453.

[h] Single precipitation by acid digestion and cinchonine. Tungsten corrected for silicon, iron, chromium, vanadium, and molybdenum.

[i] ZnO separation.

[j] Tungsten removed and phosphorus precipitated as the molybdate in hot, strong nitric acid solution.

[k] Nitric-hydrochloric acid dehydration.

[l] Glyoxime precipitate ignited and weighed as NiO.

[m] Ferrous sulfate-persulfate-$KMnO_4$ titration method.

[n] Precipitated with H_2S in tartaric or citric acid solution, purified, and weighed as MoO_3.

[o] Bismuthate-arsenite method.

[p] Titrating solution standardized by use of a standard steel.

[q] KI-$Na_2S_2O_3$ titration.

[r] Solution in HCl, digestion with HNO_3 and precipitation with cinchonine. Precipitate dissolved in NH_4OH and tungsten precipitated with acid and cinchonine. Ignited oxide corrected for silicon, iron, chromium, vanadium, and molybdenum.

[s] Glyoxime precipitate titrated with standard KCN.

[t] Perchloric acid oxidation.

[u] Chromium volatilized as CrO_2Cl_2.

[v] Combustion method. L. P. Chase, Carnegie-Illinois Steel Corporation, South Works, Chicago, Ill., and T. S. Woodward, Carnegie-Illinois Steel Corporation, Ohio Works, Youngstown, Ohio, reported, respectively, 0.006 and 0.003 percent of sulfur by the combustion method.

[w] Tungsten precipitated by digestion with aqua regia and removed by filtration. Molybdenum precipitated in the filtrate with α-benzoinoxime, precipitate dissolved by digestion with HNO_3 and $HClO_4$. Solution treated with NH_4OH, filtered, and molybdenum precipitated as $PbMoO_4$, ignited, and weighed.

[x] Chromium separated as $PbCrO_4$.

[y] Perchloric acid dehydration.

[z] Differential titration using $KMnO_4$ and o-phenanthroline-ferrous complex indicator.

[z1] Major portion of tungsten precipitated from a 1-g sample by fuming with $HClO_4$ and HF. Molybdenum and remaining tungsten precipitated with α-benzoinoxime. Combined precipitates ignited to constant weight at 520° C and weighed. Oxides then heated at 750° to 800° C for 1 hour and weighed. The difference in weights represents the molybdenum volatilized. Residue analyzed for NaOH insoluble and molybdenum, and tungsten determined by difference.

[z2] Sample treated with HCl-HNO_3. Perchloric acid added and solution fumed, diluted, and filtered. Phosphorus precipitated in hot, strong nitric acid solution. Phosphomolybdate titrated with alkali.

[z3] Titrated with standard KCN solution.

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The steel for the preparation of this standard was furnished by the Climax Molybdenum Co.

WASHINGTON, September 24, 1942.

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U. S GOVERNMENT PRINTING OFFICE 492450