U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

STANDARD SAMPLE 134

MOLYBDENUM—TUNGSTEN—CHROMIUM—VANADIUM STEEL

ANALYST*	C	Mn	P	S	Si			Cr	V	Mo		W	
	Direct combustion	Persulfate-Arsenite	Alkali-Molybdate [a]	Gravimetric (direct oxidation and final precipitation of iron (+ r reduction of iron)	Nitric-sulfuric acid dehydration	COPPER H_2S–CuS–CuO	NICKEL Weighed as nickel dimethylglyoxime	$FeSO_4$–$KMnO_4$ titration	HNO_3 oxidation, potentiometric titration in presence of tungsten	Gravimetric	Colorimetric		
1	0. 808	[b]0. 152	[c]0. 014	0. 005	[d]0. 324	[e]0. 113	0. 075	[f]3. 74	1. 12	[g]8. 74		[h]1. 82	
2	. 810	[i]. 148	[j]. 016	. 005	[k]. 32	[l]. 108	[m]. 078	3. 72	1. 14	[n]8. 63	8. 70	[o]1. 76	
3	. 820	[p]. 145	[j]. 013	[q]. 009	[d]. 323	[e]. 120	[r]. 08	3. 70	1. 12	[g]8. 64		1. 84	
4	. 80	[s]. 17	[t]. 017	. 005	[u]. 32	. 12	[m]. 087	3. 76	[v]1. 09	[n]8. 60		1. 85	
5	. 817	[s]. 153			[u]. 335		[m]. 08	3. 75	1. 16	[n]8. 81		[h]1. 82	
6	. 808	[w]. 141	. 017	. 003	[d]. 319	[x]. 111	. 070	[f]3. 70	1. 10	[n]8. 61	8. 58	[y]1. 79	
7	. 808	. 148	[j]. 015	. 007	. 32	[s]. 114	. 075	3. 74	1. 12	[g]8. 80		[h]1. 85	
8	. 806	. 16	. 017	. 007	. 314	. 116	. 081	3. 76	1. 16	8. 78		1. 79	
9	. 811	[b]. 166	. 012	. 004	[d]. 329	. 118	. 071	[f]3. 73	1. 12	[z1]8. 69		[h]1. 85	
)								[f]3. 72	1. 15	[z2]8. 60		[z2]1. 78	
x1	. 814	. 16	[t]. 016	. 004	[k]. 345			[z3]3. 75	[v]1. 16	[n]8. 70		1. 91	
12	. 814	[p]. 157	. 016	[q]. 010	[u]. 32	[e]. 114	[m]. 083	3. 73	1. 13	[n]8. 69		[z4]1. 80 [h]1. 82	
13	. 810	[s]. 16	[t]. 018	. 008	[u]. 308	[z5]. 110	[r]. 070	[z6]3. 71	[v]1. 10		8. 70	[h]1. 81	
Averages	**0. 810**	**0. 155**	**0. 016**	**0. 006**	**0. 323**	**0. 114**	**0. 077**	**3. 73**	**1. 13**	**8. 69**	**8. 66**	**1. 82**	
General avg.	**0. 810**	**0. 155**	**0. 016**	**0. 006**	**0. 323**	**0. 114**	**0. 077**	**3. 73**	**1. 13**	**8. 68**		**1. 82**	

[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 $Mg_2P_2O_7$. (Phosphorus not detected in tungstic acid residue.)

[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] Chromium volatilized as CrO_2Cl_2.

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

[k] Perchloric acid dehydration.

[l] Ammonia-copper-complex colorimetric method.

[m] Glyoxime precipitate ignited and weighed as NiO.

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

[o] Tungsten separated by acid hydrolysis and cinchonine, purified by carbonate fusion, and finally precipitated with $HgNO_3$. Ignited oxide corrected for molybdenum and vanadium.

[p] Chromium separated with ZnO. Finished by bismuthate-arsenite method.

[q] Combustion.

[r] Glyoxime precipitate titrated with KCN solution.

[s] Chromium separated with ZnO.

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

[u] Nitric-hydrochloric acid dehydration.

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

[w] Bismuthate-arsenite.

[x] KI–$Na_2S_2O_3$ titration.

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

[z] H_2S—α-benzoinoxime—CuO.

[z1] Molybdenum precipitated as MoS_3 from tartaric acid solution, precipitated with α-benzoinoxime, ignited, and weighed as MoO_3.

[z2] 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.

[z3] Permanganate oxidation.

[z4] Hydroquinone colorimetric method. See Z. Anal. Chem. **114**, 170 (1938).

[z5] Copper precipitated as CuCNS. Finished by KI-$Na_2S_2O_3$ titration.

[z6] Chromium oxidized by phosphoric-perchloric acid mixture.

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