U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE 101A

18 CHROMIUM—8 NICKEL STEEL

	C	Mn	P		S		Si		Ni	Cr					
*ANALYST**	*Direct combustion 1,300° to 1,375° C*		*Gravimetric (Weighed as $Mg_2P_2O_7$ after removal of arsenic)*	*Alkali-molybdate* [a]	*Gravimetric (Direct oxidation and final precipitation in reduced solution)*	*Evolution with HCl ZnS-Iodine (theoretical sulfur titre)* [a]	*Perchloric acid dehydration*	*COPPER H_2S-CuS-CuO*	*Weighed as nickel dimethylglyoxime*	*$FeSO_4$-$KMnO_4$ titration*	*VANADIUM*	*MOLYBDENUM (colorimetric)*	*COBALT*	*NITROGEN*	
1	0. 047	0. 466 [c]	0. 017	0. 019	0. 008	0. 010	0. 338 [d]	0. 050 [e]	8. 98	18. 33 [f]	0. 029 [g]	0. 012	0. 071 [h]	0. 044 [i]	
2	. 046 [j]	. 47 [k]		. 016		. 008	. 333 [d]	. 05	9. 02 [l]	18. 33					
3	. 049	. 473 [k]		. 016	. 008	. 009	. 344 [d]	. 046 [e]	8. 95	18. 32				. 046 [m]	
4	. 051 [j]	. 460 [n]		. 014	. 008	. 010	. 341 [d]	. 053 [e]	8. 98 [l]	18. 30 [o]				. 044	
5	. 047	. 469 [c]	. 016	. 017		. 010	. 349	. 059	9. 00	18. 30 [f]	. 027 [g]	. 010	. 062 [p]		
6	. 052	. 460 [k]		. 016		. 009	. 331	. 050 [q]	8. 94	18. 35					
7	. 052 [j]	. 45 [r]		. 021	. 010	. 012	. 333 [d]	. 054	8. 98 [l]	18. 34				. 043 [s]	
8	. 045 [j]	. 466 [t]		. 018		. 009	. 330	. 054	8. 97 [l]	18. 31 [o]	. 038 [u]	. 011	. 080 [v]	. 045 [w]	
9	. 048 [j]	. 466 [x]	. 018	. 019	. 011	. 010 [y]	. 339 [d]	. 049	9. 01	18. 36	. 040 [z]	. 008	. 065 [v]		
10	. 048 [j]	. 474 [x]	. 014	. 015	. 010	. 011	. 337	. 049 [e]	9. 02	18. 36	.040 [z1]	. 009	. 060 [v]		
11	. 051 [j]	. 464 [x]	. 017	. 018	. 008	. 009	. 344 [d]	. 046	8. 96	18. 32					
12	.049 [z2]	. 460		. 021			. 339		9. 02 [l]	18. 34					
Averages	**0. 049**	**0. 465**	**0. 016**	**0. 018**	**0. 009**	**0. 010**	**0. 338**	**0. 051**	**8. 99**	**18. 33**	**0. 034**	**0. 010**	**0. 068**	**0. 044**	
Recommended values	**0. 049**	**0. 465**	**0. 017**		**0. 009**		**0. 338**	**0. 051**	**8. 99**	**18. 33**	**0. 030**	**0. 010**	**0. 070**	**0. 044**	

[a] Precipitated at 40° C, washed with a 1-percent solution of KNO_3 and titrated with alkali standardized by using the National Bureau of Standards Standard Sample of acid potassium phthalate and the ratio 23 NaOH:1 P.

[b] Value obtained by standardizing the titrating solution by means of sodium oxalate through $KMnO_4$ and $Na_2S_2O_3$.

[c] Bismuthate ($FeSO_4$-$KMnO_4$) method after ZnO separation.

[d] Double dehydration.

[e] Finished by electrolysis.

[f] Persulfate oxidation, potentiometric titration with $FeSO_4$ standardized with $K_2Cr_2O_7$.

[g] 10-g sample dissolved in 120 ml of diluted HCl(1+2). Sufficient ZnO added to precipitate all the chromium and vanadium. Solution filtered and the precipitate dissolved in diluted HNO_3. 0.6-g of $Na_2HPO_4.12H_2O$ added, and phosphorus and vanadium precipitated with ammonium molybdate. Solution filtered and the precipitate dissolved in H_2SO_4-HNO_3 and evaporated to fumes. Solution diluted, treated with SO_2 to reduce any oxidized chromium; vanadium then oxidized with HNO_3 and titrated potentiometrically with $FeSO_4$.

[h] 10-g sample dissolved in diluted HCl(1+1). Bulk of the iron separated by extraction with ether. Residual iron and chromium in extracted acid-portion separated from cobalt by double precipitation with ZnO. Cobalt precipitated twice with α-nitroso-β-naphthol, ignited, and weighed as Co_3O_4.

[i] Determination made by Vernon C. Holm, by the vacuum-fusion method. See BS J. Research 7, 375 (1931) RP346.

[j] Burned with tin at 1,100° to 1,300° C.

[k] Persulfate-arsenite method after ZnO separation.

[l] Titrated with standard KCN solution.

[m] Solution-distillation (Allen) method. Sample dissolved in diluted HCl(1+1).

[n] Bismuthate-arsenite method after ZnO separation.

[o] Perchlorate acid oxidation.

[p] Bulk of iron removed with ether. Chromium separated as $PbCrO_4$ from perchloric acid solution. Solution treated with cupferron, and cobalt precipitated in filtrate with α-nitroso-β-naphthol and weighed as Co_3O_4.

[q] KI-$Na_2S_2O_3$ titration.

[r] Persulfate-arsenite in presence of chromium. See Iron Age **142**, No. 26; 16 (1938).

[s] Average value obtained by the solution-distillation method after solution of sample in (1) diluted HCl(1+1), (2) diluted H_2SO_4(1+3), and (3) diluted HCl (1+1), followed by addition of $HClO_4$ and evaporation to fumes of $HClO_4$.

[t] Bismuthate ($FeSO_4$-$KMnO_4$) method after separation of chromium as $PbCrO_4$.

[u] Chromium separated as $PbCrO_4$. Vanadium determined by differential titration with $FeSO_4$-$KMnO_4$ using o-phenanthroline indicator. See Sampling and Analysis of Carbon and Alloy Steels, Chemists of the U. S. Steel Corporation, p. 160-161 (1938).

[v] ZnO-α-nitroso-β-naphthol method. Precipitate ignited and weighed as Co_3O_4.

[w] Solution-distillation method. Sample dissolved in sulfuric acid.

[x] Persulfate-arsenite method after removal of chromium as CrO_2Cl_2. (See Ind. Eng. Chem., Anal. Ed. **10**, 360 (1938).

[y] Sample ignited in oxygen, gasses passed into H_2O_2 and H_2SO_4 titrated.

[z] Mercury cathode separation. Vanadium reduced with SO_2, and titrated with $KMnO_4$ after removal of excess SO_2. See reference in footnote u, p. 164-165.

[z1] $FeSO_4$-$KMnO_4$ titration after removal of chromium as CrO_2Cl_2. See reference in footnote u, p. 157 160.

[z2] Burned with Pb_3O_4 at 1,300° C.

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