U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE 121

18 CHROMIUM—8 NICKEL STEEL

(TITANIUM-BEARING)

ANALYST*	C	Mn	P		S		Si		Ni	Cr	Ti		V	Mo	Co	
	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) [b]	Perchloric acid dehydration	COPPER H_2S-CuS-CuO	Weighed as nickel dimethylglyoxime	$FeSO_4$-$KMnO_4$ titration	GRAVIMETRIC	Colorimetric	VANADIUM	MOLYBDENUM (Colorimetric)	COBALT	
1	0. 058	0. 413 [c]	0. 017		0. 004		0. 371 [d]	0. 045 [e]	9. 05	17. 85 [f]	0. 395 [g]		0. 032 [h]	0. 011	0. 075 [i]	
2	. 061	. 415 [j]	. 014	. 013	. 008		. 366	. 039 [k]	9. 08	17. 89	. 393 [g]					
3	. 055 [l]	. 40 [m]		. 012	. 006	. 004	. 367	. 042 [e]	9. 01	17. 78 [f]		. 394	. 052 [h]			
4	. 058 [l]	. 415 [j]		. 015		. 005	. 382		9. 05	17. 85	. 39 [g]					
5		. 405 [c]		. 016	. 007 [n]				9. 05	17. 83 [f]	. 392 [g]	. 405	. 048 [h]			
6	. 058 [l]	. 41					. 38 [d]	. 050	9. 00 [o]	17. 76	. 377 [p]					
7	. 056 [q]	. 40 [j]		. 018	. 007 [r]	. 008 [s]	. 36	. 045	9. 05	17. 93	. 387		. 038			
8	. 057	. 407 [u]		. 016		. 006 [t]	. 368 [v]	. 041	9. 04 [o]	17. 82 [f]		. 40	. 054	. 011	. 080	
9	. 053	. 413 [u]	. 015	. 013	. 006 [n]	. 006 [w]	. 372	. 048 [k]	9. 04	17. 78	. 394 [g]				. 076 [x]	
10	. 058 [l]	. 416 [c]		. 017		. 005	. 371	. 045 [k]	9. 07 [o]	17. 84		. 394		. 009		
11	. 056 [l]	. 413 [c]		. 015		. 004	. 366	. 045	9. 05	17. 82 [y]		. 39	. 035 [z]	. 008	. 09 [z1]	
12	. 054 [l]	. 406 [z2]	. 013	. 015	. 004	. 004	. 372	. 044	9. 05	17. 85		. 398	. 038 [z3]	. 008	. 076 [z1]	
13	. 059	. 410 [z2]	. 014	. 012	. 009	. 007 [w]	. 378	. 045	9. 00	17. 80 [y]	. 402 [g]					
14	. 054 [z4]	. 418 [z2]	. 017	. 018	. 009 [n]	. 010 [t]	. 371 [d]	. 050	9. 07 [o]	17. 81	. 39 [g]	. 40		. 010	. 076 [z1]	
15	. 058 [q]	. 403 [j]		. 018			. 365		9. 01 [o]	17. 81		. 404				
16	. 061 [z5]	. 409 [j]		. 016		. 004 [w]	. 373 [d]		9. 07	17. 88		. 39				
17	. 057 [z6]	. 405 [z7]	. 015	. 012			. 368 [d]		9. 03	17. 81		. 39				
ages	. 057	. 409	. 015	. 015	. 007	. 005	. 371	. 045	9. 04	17. 83	. 391	. 397	. 042	. 010	. 080	
Recommended values	0. 057	0. 409	0. 016		0. 096		0. 371	0. 045	9. 04	17. 83	0. 394		0. 035	0. 010	0. 08	

[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] Persulfate-arsenite method after removal of chromium as $PbCrO_4$ from perchloric acid solution.

[d] Double dehydration.

[e] Finished by electrolysis.

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

[g] Solution in diluted H_2SO_4 and titanium precipitated with cupferron. Precipitate filtered and ignited, treated with $HClO_4$-HF, taken to dryness, ignited and fused with $Na_2S_2O_7$. Melt dissolved in diluted H_2SO_4, treated with an excess of NaOH, filtered and precipitate dissolved in diluted H_2SO_4. (Alternately, the ignited precipitate treated with H_2SO_4-HF, fumed, and the residue filtered, ignited, fused with $K_2S_2O_7$, and the melt dissolved in the main solution.) H_2S group removed, and residual iron precipitated in ammoniacal tartrate solution with H_2S. Filtrate acidified and titanium precipitated with cupferron. Ignited TiO_2 corrected for V_2O_5 and SiO_2. Analyst 5 also corrected for 0.003 percent of ZrO_2. (See Ind. Eng. Chem., Anal. Ed. **5**, 305 (1933).)

[h] Nitric acid oxidation, potentiometric titration with $FeSO_4$ solution standardized against $K_2Cr_2O_7$.

[i] Cobalt separated from iron and chromium by ether separation followed by double ZnO separation, then precipitated twice with α-nitroso-β-naphthol, ignited and weighed as Co_3O_4.

[j] Persulfate-arsenite method after ZnO separation.

[k] KI-$Na_2S_2O_3$ titration.

[l] Burned with tin at 1,100° to 1,250° C.

[m] Bismuthate oxidation, potentiometric titration with $HgNO_3$ solution.

[n] Meineke's method.

[o] Titrated with standard KCN solution.

[p] Titanium separated from most of the iron (ferrous) by precipitation with cupferron. Precipitate ignited and fused with Na_2CO_3. Solution, of melt acidified, treated with an excess of Na_2O_2 and boiled. Precipitate filtered, dissolved in acid solution treated with SO_2, and nearly neturalized with NH_4OH. Titanium then precipitated by hydrolysis with $Na_2S_2O_3$. Precipitate ignited, treated with H_2SO_4-HF, ignited and weighed as TiO_2.

[q] Burned with Pb_3O_4 at 1,250° to 1,350° C.

[r] Iron, chromium, and nickel removed by use of the mercury cathode. Finished gravimetrically as $BaSO_4$.

[s] Sample ignited in oxygen, gases passed into alkaline peroxide and sulfur precipitated as $BaSO_4$.

[t] Sample ignited in oxygen, gases passed into H_2O_2, and H_2SO_4 titrated.

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

[v] Double dehydration with sulfuric acid.

[w] Solution in HCl (1+1).

[x] Bulk of iron removed with ether. Chromium separated as $PbCrO_4$ from perchloric acid solution. H_2S group removed, remaining iron and chromium removed by NH_4OH and ZnO. Cobalt precipitated with α-nitroso-β-naphthol, ignited and weighed as Co_3O_4.

[y] Perchloric acid oxidation.

[z] Bulk of chromium removed as $PbCrO_4$. Differential titration with $KMnO_4$ and ortho phenanthroline ferrous iron indicator. (See Ind. Eng. Chem., Anal. Ed. **6**, 48 (1934).)

[z1] ZnO-α-nitroso-β-naphthol method. Weighed as Co_3O_4.

[z2] Persulfate-arsenite method after volatilization of chromium as CrO_2Cl_2. (See Ind. Eng. Chem., Anal. Ed. **10**, 360 (1938).)

[z3] Titrated with standard $KMnO_4$ after removal of chromium as CrO_2Cl_2.

[z4] Burned with copper oxide and tin at 1,300° C.

[z5] Burned with magnesium at 1,100° C.

[z6] Burned with ingot iron at 1,250° C.

[z7] Chromium separated by precipitating with $NaHCO_3$ and manganese then determined by bismuthate ($FeSO_4$-$KMnO_4$) method.

*LIST OF ANALYSTS

1. Ferrous Laboratory, National Bureau of Standards, Analysis by John L. Hague and Waino H. Jukkola.
2. D. P. Bartell, Allegheny Steel Co., Brackenridge, Pa.
3. J. A. Wiley, The Midvale Co., Philadelphia, Pa.
4. H. N. Austin, The Babcock & Wilcox Tube Co., Beaver Falls, Pa.
5. T. R. Cunningham and R. M. Fowler, Union Carbide and Carbon Research Laboratories, Niagara Falls, N. Y.
6. C. M. Johnson, The Crucible Steel Company of America, Parks Works, Pittsburgh, Pa.
7. Chemical Laboratory of the Timken Steel and Tube Division, The Timken Roller Bearing Co., Canton, Ohio.
8. W. Dillon, The Carpenter Steel Co., Reading, Pa.
9. Armco Research Chemical Laboratory, A. H. Thomas, in charge; analysis by O. B. Ellis and S. A. Lapham, Middletown, Ohio.
10. W. C. Maurer, Carnegie-Illinois Steel Corporation, Wood Works, McKeesport, Pa.
11. W. D. Brown, Carnegie-Illinois Steel Corporation, Duquesne Works, Duquesne, Pa.
12. W. F. Muehlberg, American Steel and Wire Co., Cleveland District Laboratory, Cleveland, Ohio.
13. W. M. Davidson, National Tube Co., Ellwood Works, Ellwood City, Pa.
14. L. P. Chase, Carnegie-Illinois Steel Corporation, South Works, Chicago, Ill.
15. G. P. Burks, Carnegie-Illinois Steel Corporation, Gary Works, Gary, Ind.
16. S. S. Heide, Tennessee Coal, Iron and Railroad Co., Ensley, Ala.
17. A. H. Ward, Columbia Steel Company, Pittsburg Works, Pittsburg, Calif.

Washington, D. C.
January 10, 1939.

LYMAN J. BRIGGS,
Director.

U. S. GOVERNMENT PRINTING OFFICE 131480