

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		VANADIUM	MOLYBDENUM (Colorimetric)	COBALT	
	Direct combustion 1,300° to 1,375° C.		Gravimetric (Weighed as Mg ₂ P ₂ O ₇ after re- moval of arsenic)	Alkali-molybdate ^a	Gravimetric (Direct oxidation and final precipitation in re- duced solution)	Evolution with HCl ZnS-Iodine (theo- retical sulfur titre) ^b	Perchloric acid dehy- dration	COPPER H ₂ S-CuS-CuO	Weighed as nickel di- methylglyoxime	FeSO ₄ -KMnO ₄ titra- tion	GRAVIMETRIC				Colorimetric
1	0.058	0.413 ^c	0.017		0.004		0.371 ^d	0.045 ^e	9.05	17.85	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		.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	.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 ^u	.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 ^v		.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 ^v	.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			
Averages	.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		0.006		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 KNO₃ 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 KMnO₄ and Na₂S₂O₃.

^c Persulfate-arsenite method after removal of chromium as PbCrO₄ from perchloric acid solution.

^d Double dehydration.

^e Finished by electrolysis.

^f Persulfate oxidation, potentiometric titration with FeSO₄ solution standardized against K₂Cr₂O₇.

^g Solution in diluted H₂SO₄ and titanium precipitated with cupferron. Precipitate filtered and ignited, treated with HClO₄-HF, taken to dryness, ignited and fused with Na₂S₂O₇. Melt dissolved in diluted H₂SO₄, treated with an excess of NaOH, filtered and precipitate dissolved in diluted H₂SO₄. (Alternately, the ignited precipitate treated with H₂SO₄-HF, fumed, and the residue filtered, ignited, fused with K₂S₂O₇, and the melt dissolved in the main solution.) H₂S group removed, and residual iron precipitated in ammoniacal tartrate solution with H₂S. Filtrate acidified and titanium precipitated with cupferron. Ignited TiO₂ corrected for V₂O₅ and SiO₂. Analyst 5 also corrected for 0.003 percent of ZrO₂. (See Ind. Eng. Chem., Anal. Ed. 5, 305 (1933).)

^h Nitric acid oxidation, potentiometric titration with FeSO₄ solution standardized against K₂Cr₂O₇.

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 Co₂O₃.

^j Persulfate-arsenite method after ZnO separation.

^k KI-Na₂S₂O₃ titration.

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

^m Bismuthate oxidation, potentiometric titration with HgNO₂ solution.

ⁿ Meisner'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 Na₂CO₃. Solution, of melt acidified, treated with an excess of Na₂O₂ and boiled. Precipitate filtered, dissolved in acid solution treated with SO₂, and nearly neutralized with NH₄OH. Titanium then precipitated by hydrolysis with Na₂S₂O₃. Precipitate ignited, treated with H₂SO₄-HF, ignited and weighed as TiO₂.

^q Burned with Pb₂O₄ at 1,250° to 1,350° C.

^r Iron, chromium, and nickel removed by use of the mercury cathode. Finished gravimetrically as BaSO₄.

^s Sample ignited in oxygen, gases passed into alkaline peroxide and sulfur precipitated as BaSO₄.

^t Sample ignited in oxygen, gases passed into H₂O₂, and H₂SO₄ titrated.

^u Bismuthate (FeSO₄-KMnO₄) 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 PbCrO₄ from perchloric acid solution. H₂S group removed, remaining iron and chromium removed by NH₄OH and ZnO. Cobalt precipitated with α-nitroso-β-naphthol, ignited and weighed as Co₂O₃.

^y Perchloric acid oxidation.

^z Bulk of chromium removed as PbCrO₄. Differential titration with KMnO₄ and ortho phenantrolone ferrous iron indicator. (See Ind. Eng. Chem., Anal. Ed. 6, 48 (1934).)

^{z1} ZnO-α-nitroso-β-naphthol method. Weighed as Co₂O₃.

^{z2} Persulfate-arsenite method after volatilization of chromium as CrO₂Cl₂. (See Ind. Eng. Chem., Anal. Ed. 10, 360 (1938).)

^{z3} Titrated with standard KMnO₄ after removal of chromium as CrO₂Cl₂.

^{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 NaHCO₃ and manganese then determined by bismuthate (FeSO₄-KMnO₄) method.

*LIST OF ANALYSTS

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