

U. S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE 126

HIGH-NICKEL STEEL

(Furnished by Carpenter Steel Company)

ANALYST*	Ni		C	Mn		Si	COPPER H ₂ S-CuS-CuO	COBALT	CHROMIUM
	Weighted as nickel dimethylglyoxime	Electrolysis	Cyanide titration	Direct combustion	Bismuthate (FSO ₄ -KMnO ₄)	Persulfate-arsenite			
1	^a 36.43	^b 36.43		0.035		0.504	^e 0.105	^d 0.007	^e 0.008
2	36.45			.038	0.508	.505	.111	^f .09	^g .009
3	^a 36.44	^h 36.48		.032		.502	.104	ⁱ .096	ⁱ .008
4		^j 36.46		.034	.502		.096	^f .092	^k .01
5		^l 36.46		.032	.506		.109	^f .094	^m .01
6	ⁿ 36.40		^o 36.38	.037	.509		.115	.105	^k .01
7	^q 36.43	^j 36.41		.032		.502	.109	.092	^s .009
8	^t 36.48	^u 36.49		.028		.508	^p .113	^f .093	^v .008
9	^w 36.39			.036		.499	.109	^f .096	^x .006
10	36.35			.031		.502	.116	.103	
		^y 36.46		.032	.512	.513	^p .112	^f .099	^k .007
Averages	36.42	36.46	36.38	0.034	0.507	0.504	0.109	0.095	0.008
Recommended values	36.42			0.034	0.506		0.109	0.095	0.008

^a Double precipitation, using a 0.25-g sample. With a single precipitation on a 0.2-g sample, a value of 36.47 percent of nickel was obtained by analyst 1.

^b Nickel separated from iron by glyoxime on a 1-g sample, and then deposited electrolytically on a platinum-gauze cathode in ammoniacal solution with a current of 0.2 amp/dm² for about 6 hours. Result corrected for a positive blank (0.0003 g) obtained by electrolysis of 0.3000 g of 99.99-percent nickel. By reversing the polarity and reprecipitating the first plate from a fresh electrolyte, a value of 36.41 percent of nickel was obtained. Traces of nickel were recovered with glyoxime in both electrolyses.

^c Double dehydration with sulfuric acid.
^d Cobalt separated from iron by ether separation followed by a double ZnO separation, then precipitated with α -nitroso- β -naphthol. Ignited precipitate dissolved, acid solution treated with H₂S, filtered, cobalt precipitated with α -nitroso- β -naphthol and weighed as Co₂O₄. (See BS J. Research 7, 883 (1931) RP380.)

^e Silver nitrate-persulfate oxidation.
^f Finished by electrolysis.
^g Double ZnO, α -nitroso- β -naphthol. Precipitate ignited and weighed as Co₂O₄.

^h Nickel precipitated by glyoxime on a 0.25-g sample. Solution filtered, precipitate dissolved, two such solutions (=0.5-g sample) combined and nickel deposited by electrolysis.

ⁱ Double precipitation with ZnO. Combined filtrates evaporated to 50 ml and cobalt precipitated twice with KNO₃. Precipitate ignited, dissolved, solution treated with sodium acetate, and filtered.

Cobalt precipitated as sulfide in the filtrate. Precipitate ignited and converted to CoSO₄.

^j Ether separation—electrolysis on a 1-g sample.
^k Ether separation—cupferron— α -nitroso- β -naphthol method. (See BS J. Research 8, 659 (1932) RP445.)

^l Bulk of the iron removed with ether on a 5-g sample. Extracted acid solution treated with H₂S to remove copper. Residual iron and manganese separated with ammonia and ammonium persulfate. Filtrate electrolyzed and weight of nickel corrected for cobalt.

^m Ether separation on a 5-g sample. Extracted acid solution evaporated to dryness and cobalt twice separated as potassium cobaltinitrite and cobalt finally weighed as CoSO₄.

ⁿ One-gram sample dissolved in diluted HNO₃ (1+1). Nickel precipitated with glyoxime in a weighed aliquot portion equal to an approximately 0.1-g sample. Solution filtered on a glass-frit crucible, precipitate dried at 110° C, weighed, and dissolved in dilute acid. Glyoxime destroyed, and traces of Fe₂O₃ precipitated with ammonia. Solution filtered, and ignited precipitate weighed, multiplied by the nickel-glyoxime factor (0.2032), and the equivalent percentage of nickel (0.04%) subtracted from the nickel indicated by the first glyoxime precipitate.

^o Photometric titration, see Ind. Eng. Chem., Anal. Ed. 10, 175 (1938).

^p Double dehydration.
^q Single precipitation on a 0.1-g sample.
^r Double dehydration with hydrochloric acid.

^s Ether separation, copper removed with H₂S. Cobalt twice precipitated with KNO₃. Residue, iron removed by a basic acetate separation. Cobalt then precipitated with colorless (NH₄)₂S and weighed as Co₂O₄.

^t Single precipitation on a 0.5-g sample, with recovery of traces of nickel (=0.031 Ni) in the glyoxime filtrate.

^u Iron separated on a 1-g sample by double precipitation with ammonia and ammonium persulfate. Filtrate acidified with sulfuric acid, and evaporated to fumes of sulfuric acid. Solution diluted and nickel deposited by electrolysis in ammoniacal solution. Corrections made for copper, cobalt, blank (see treatment h), and for nickel recovered in the electrolyte and ammonia precipitate.

^v Ether separation—H₂S-ZnO- α -nitroso- β -naphthol. Cobalt weighed as Co₂O₄.

^w Single precipitation on 0.25-g sample.
^x Double ZnO separation. Cobalt twice precipitated with α -nitroso- β -naphthol and weighed as Co₂O₄.

^y Nickel first separated from iron by glyoxime on a 2-g sample.

Analyst 1 also reported 0.002 percent of vanadium, and analyst 6, 0.014 percent of sulfur. In addition, spectrochemical examination by B. F. Scribner indicated traces of tin, aluminum, calcium, and molybdenum. Calcium was not detected chemically by analyst 1 in tests on a 5-g sample by a procedure sensitive to 0.005 percent of calcium. Analyst 6 also found no calcium by a method in which a solution of a 10-g sample was electrolyzed in a mercury cathode cell and the electrolyte finally tested with oxalate.

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Washington, D. C.
December 7, 1939

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Director.