

UNITED STATES DEPARTMENT OF COMMERCE
WASHINGTON 25, D. C.

National Bureau of Standards
Certificate of Analyses
Standard Sample 65d
Basic Electric Steel, 0.3% Carbon

ANALYST	C	Mn	P		S			Si	Cu	Ni	Cr	V	Mo	Al		Sn	N
	Persulfate-Arsenite	Gravimetric (weighed as $Mg_2P_2O_7$ after removal of arsenic)	Alkali-Molybdate ^a	Gravimetric (direct oxidation and precipitation after reduction of iron)	Combustion iodate titration	Evolution with HCl (1+1) ZnS-Iodine (theoretical sulfur titer) ^b	Perochloric acid dehydration	$H_2S-CuS-CuO$	Weighted as nickel dimethylglyoxime	$FeSO_4-KMnO_4$ titration	Colorimetric	Total	Acid-insoluble (calculated as Al_2O_3)	Disillation-titration			
1	0.265	0.726	0.014	0.013	0.009	0.009	0.368	0.053	0.059	0.050	0.001	0.025	0.010	0.003	0.013		
2	.262	.728	.015	.011	.008	.011	.370	.050	.061	.048	.002	.027	.008	.004	.013		
3	.262	.728	.015	.011	.008	.011	.370	.051	.059	.047	.001	.028	.008	.004	.012		
4	.261	.732	.015	.010	.010	.012	.376	.049	.061	.049	.001	.024	.008	.004	.012		
5	.261	.732	.012	.013	.009	.009	.368	.049	.059	.049	.002	.025	.009	1.004	h1.012		
6	.266	.721	.015	.011	.011	.010	.374	.048	.061	.050	.001	.023	.004	.004	h1.012		
7	.262	.726	.016	.016	.010	.009	.362	.049	.060	.044	.002	.023	.005	.007	.013		
8	.267	.733	.016	.016	.010	.011	.378	.052	.060	.052	.003	.025	.006	1.006	m.013		
9	.267	.738	.014	.009	.009	.011	.362	.054	.058	.046	.002	.028	.007	.014	.013		
Average	0.264	0.730	0.015	0.010	0.010	0.010	0.370	0.051	0.060	0.049	0.002	0.025	0.009	0.004	0.013		
General average	0.264	0.730	0.015	0.010	0.010	0.010	0.370	0.051	0.060	0.049	0.002	0.025	0.009	0.004	0.013		

^a Precipitated at 40° 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 23NaOH:1P.
^b Value obtained by standardizing the titrating solution by means of sodium oxalate through $KMnO_4$ and $Na_2S_2O_3$ and use of the ratio 2I:1S.
^c Potentiometric titration.
^d Molybdenum-blue photometric method. See J. Research NBS 26, 405 (1941) RP1386.
^e 1-g sample burned in oxygen at 1,425° C and sulfur dioxide absorbed in starch-iodide solution. Iodine liberated from iodide by titration, during the combustion, with standard KIO_3 solution based on 93 percent of the theoretical factor.
^f Double dehydration with intervening filtration.
^g Diethyldithiocarbamate photometric method. See J. Research NBS 47, 380 (1951) RP2265.
^h Chromium separated from the bulk of the iron in a 10-g sample by hydrolytic precipitation with $NaHCO_3$, oxidized with persulfate, and titrated potentiometrically with ferrous ammonium sulfate.
ⁱ Vanadium separated as in (h), oxidized with HNO_3 and titrated potentiometrically with ferrous ammonium sulfate.
^j Most of the iron in a 1-g sample removed by a mercury

cathode separation. Cupferron-chloroform extraction of the electrolyte at pH 3.5. Extracted cupferrates converted to perchlorates. Second cupferron-chloroform extraction made at 4N acidity to remove iron, vanadium, etc. Aluminum, in the aqueous layer, determined by the aluminum method.
^k 10-g sample treated with dilute HNO_3 (1+3). Aluminum determined in the separated insoluble residue as in (j), omitting the mercury cathode separation.
^l Sulfide-iodine method. See BS J. Research 8, 309 (1932) RP415.
^m Sulfuric acid digestion for 3 hr of a 1-g sample. See J. Research NBS 43, 201 (1949) RP2021.
ⁿ Titrating solution standardized with a standard steel.
^o Sulfuric acid dehydration.
^p Copper-ammonia-complex photometric method.
^q Dimethylglyoxime photometric method.
^r Diphenylglyoxime photometric method.
^s $NaHCO_3-FeSO_4-(NH_4)_2S_2O_8-KMnO_4$ method.
^t Aluminum-photometric method.
^u Stannous-iodate titration method.
^v Bismuthate method.
^w Weighed as ammonium phosphomolybdate.
^x Spectrographic determination.

^y $NaHCO_3-NaOH-8$ -hydroxyquinoline precipitation. Bromate-thiosulfate titration.
^z Gasometric method.
^{aa} Periodate photometric method.
^{ab} Persulfate oxidation, potentiometric titration with $FeSO_4$.
^{ac} Persulfate photometric method.
^{ad} $KI-Na_2S_2O_8$ titration.
^{ae} $H_2S-MoO_3-MoO_3$.
^{af} $NaHCO_3-NaOH-ALPO_3$.
^{ag} Initial mercury cathode separation. Iron and other interfering elements removed as $CHCl_3$ -soluble 8-hydroxyquinolindates. Al extracted with $CHCl_3$ as the 8-hydroxyquinolinate and determined photometrically at 389m μ . See Anal. Chem. 28, 1520 (1956).
^{ah} Finished photometrically with Nessler's reagent.
^{ai} Diethyldithiocarbamate photometric method.
^{aj} Eriochrome cyanine-R photometric method.
^{ak} $NaHCO_3$ -cupferron- $NH_4OH-Al_2O_3$.
^{al} Sulfur gases absorbed in neutral H_2O_2 and titrated with sodium borate.
^{am} Vanadium precipitated with cupferron and determined by the phosphotungstovanadate photometric method.

List of Analysts

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The steel for the preparation of this standard was furnished by the United States Steel Corporation.

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A. V. ASTIN, Director.