U.S. DEPARTMENT OF COMMERCE

National Bureau of Standards

Certificate of Analyses

OF

STANDARD SAMPLE No. 111

NICKEL-MOLYBDENUM STEEL

	C	Mn	P		S		Si		Ni			Mo		
ANALYST *	CARBON Direct combustion	MANGANESE 1. Bismuthate ($FeSO_4$-$KMnO_4$)	PHOSPHORUS 1. Alkali-molybdate)[a]	2. Gravimetric (weighed as $Mg_2P_2O_7$ after removal of arsenic)	SULPHUR 1. Gravimetric (direct oxidation and precipitation in reduced solution)	2. Evolution with HCl-ZnS - Iodine - (theoretical sulphur titre[b])	SILICON 1. Sulphuric acid dehydration	COPPER 1. H_2S-CuS-CuO	NICKEL Weighed as nickel dimethylglyoxime	CHROMIUM $FeSO_4$-$KMnO_4$ titration	VANADIUM	MOLYBDENUM 1. Gravimetric	2. Colorimetric	ARSENIC
1	0.197	0.660	0.024	0.023	0.019	0.018	0.296	0.123	1.74	0.275 [c]	0.002 [c]	0.214 [d]		0.014
2	.201	.651 [e]	.022	.023	.021	.018	.288 [f]	.133 [g]	1.73	.283 [h]	.004 [i]	.220 [j]	0.217	
3	.206	.666 [e]	.024	.023 [k]	.020	.017	.286 [f]	.120 [g]	1.76	.269	.005	.205 [j]		
4	.203	.660 [e]	.023 [l]		.021	.020 [m]	.28	.13 [g]	1.73 [n]	.28		.216 [j]	.216	
5	.20	.66	.023 [l]	.021	.019	.018	.293	.11	1.76	.26 [o]	<.001 [o]	.22 [d]		
6	.202	.67 [e]	.021			.019	.291 [f]	.127 [g]	1.76 [g]			.20 [d]	.20	
7	.207 [p]	.66 [q]	.024		.022	.022 [l]	.290	.108 [r]	1.76 [n]	.275		.22 [s]		.017
	.206	.664 [e]	.022		.018		.295	.122	1.76	.269		.211 [d]	.220	
9		.659							1.76			.216 [d]		
10	.196 [p]	.663	.022	.022	.020	.020	.300	.122 [g]	1.76 [g]	.273 [h]	.001 [t]	.216 [j]		
11	.205	.663	.024		.021	.017	.286	.125	1.75	.270 [c]	.003 [u]	.223 [v]		
12	.197	.666	.022				.306		1.73	.267		.213 [j]	.214	
Averages	**.202**	**.662**	**.023**	**.022**	**.020**	**.019**	**.292**	**.122**	**1.75**	**.272**	**.003**	**.215**	**.215**	**.016**
Recommended values	**.202**	**.662**	**.023**		**.020**		**.292**	**.122**	**1.75**	**.272**	**.003**	**.215**		**.016**

[a] Precipitated at 40° C., washed with 1 percent KNO_3 and titrated with alkali standardized by the use of National Bureau of Standards Standard Acid Potassium Phthalate and the 23:1 ratio.

[b] Value obtained by rapid solution in concentrated HCl. Titrating solution standardized against standard sodium oxalate through $Na_2S_2O_3$ and $KMnO_4$.

[c] Chromium and vanadium concentrated by hydrolysis with $NaHCO_3$ and subsequently determined by potentiometric titration.

[d] Precipitated with α-benzoinoxime and weighed as MoO_3. See B.S. Jour. Research, vol. 9 (RP 453) p. 1, 1932.

[e] Persulphate-arsenite method.

[f] Dehydration with perchloric acid.

[g] Finished by electrolysis.

[h] Chromium oxidized with $HClO_4$.

[i] Vanadium precipitated with cupferron, purified by precipitations with NaOH and cupferron and determined by reducing with SO_2 and titrating with $KMnO_4$.

[j] Weighed as $PbMoO_4$.

[k] Weighed as ammonium phosphomolybdate.

[l] Titrating solution standardized by the use of a standard steel.

[m] Sample annealed by mixing with $K_4Fe(CN)_6$ and heating at 800° C., for 20 minutes. Annealed sample dissolved in concentrated HCl, and H_2S absorbed in a solution of $CdCl_2$.

[n] Titrated with KCN. Titrating solution standardized by the use of a standard steel.

[o] Bulk of iron salted out as $FeSO_4$ in alcohol. Alcohol removed and Fe, Cr, V precipitated with Na_2CO_3. Precipitate fused with Na_2CO_3 and charcoal, extracted with H_2O, filtered and vanadium determined colorimetrically after adding H_2O_2 to the acidified filtrate.

[p] Red lead used as an accelerator.

[q] Oxidized with PbO_2 and titrated with sodium arsenite standardized by the use of standard steels.

[r] Titrated with KCN after precipitating with H_2S and igniting to oxide.

[s] Molybdenum separated with KCNS and cinchonine in reduced solution, ignited to MoO_3, and corrected for copper, cf. Iron Age, vol. 132, no. 2, p. 16 and 17, 1933.

[t] Vanadium precipitated by means of $NaHCO_3$, separated from iron, etc., by electrolysis with a mercury cathode, and estimated colorimetrically after treating with H_2O_2.

[u] Chromium and vanadium concentrated by precipitating with $NaHCO_3$, oxidized with $(NH_4)_2S_2O_8$, and titrated potentiometrically with $FeSO_4$. Vanadium then oxidized at 50 to 60° C. with $Ce(SO_4)_2$.

[v] Molybdenum precipitated with H_2S and weighed as Ag_2MoO_4.

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This standard is not recommended for colorimetric carbon determinations, because of uncertainty as to the condition of the carbon.

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