

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

National Bureau of Standards
Certificate of Analyses

Standard Sample 130A
Steel

(Lead-Bearing)

| ANALYST | C | Mn | P | | S | | | Si | Cu | Ni | Cr | V | Mo | Pb | N |
|----------------------|-------------------|--|---|-------------------------------|--|-----------------------------|---|--|---|--|--|----------------------|-------------|--|------------------------|
| | Direct combustion | Persulfate-Arsenite | Gravimetric (weighed as Mg ₂ P ₂ O 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 | Perchloric acid dehydration | Photometric | Weighed as nickel dimethylglyoxime | FeSO ₄ -KMnO ₄ titration | Photometric | Photometric | H ₂ S-PbMoO ₄ | Distillation-titration |
| 1..... | 0.177 | ° 0.758 | 0.014 | ^d 0.015 | 0.018 | ° 0.018 | 0.019 | ^f 0.172 | ^g 0.027 ^h .026 | 0.009 | ⁱ 0.012 | ^j 0.001 | 0.003 | ^k 0.229 ^l 0.227 | ^m 0.007 |
| 2..... | .184 | .745 | .015 | ^d .015 | .018 | | .019 | ^f .174 | .027 | .010 | ⁱ .012 | ° .002 | .004 | .229 | ^m .009 |
| 3..... | .179 | ° .757 | ° .017 | .017 | | ⁿ .019 | .018 | ^f .170 | ^p .029 | ^q .009 ^r .007 | ^{i,n} .011 | ^{j,n} .001 | .004 | .228 | ^m .009 |
| 4..... | .184 | .747 | .016 | ⁿ .016 | .018 | ⁿ .019 | | ^f .178 | ^h .024 | ^r .012 | .011 | ° .002 | .002 | .223 | .009 |
| 5..... | .179 | ⁿ .752 | .015 | .016 | .018 | ^s .019 | .018 | ^f .173 | ^t .028 | .008 | .013 | < .001 | .004 | .225 | .008 |
| 6..... | .185 | ⁿ .755 ^q .753 | .015 | .016 | .020 | ^u .019 | .021 | ^f .176 ^v .176 | ^w .026 | ^q .010 | ⁱ .013 | ^x .001 | .005 | .231 | ^k .008 |
| 7..... | .188 | ⁿ .752 | | .016 | | .019 | | ^f .175 | ^h .024 | .009 | .010 | ^y .002 | .004 | ^h .229 | .008 |
| 8..... | .177 | .759 | .014 | .016 | .020 | .021 | .021 | ^f .169 ^{z,f} .168 | ^t .032 | ^q .011 ^q .010 | .010 | < ^{z1} .001 | .002 | .227 | ^m .007 |
| Average..... | 0.182 | 0.753 | 0.015 | 0.016 | 0.019 | 0.019 | 0.019 | 0.173 | 0.027 | 0.010 | 0.012 | 0.001 | 0.004 | 0.228 | 0.008 |
| General average..... | 0.182 | 0.753 | 0.016 | | 0.019 | | | 0.173 | 0.027 | 0.010 | 0.012 | 0.001 | 0.004 | 0.228 | 0.008 |

^a Precipitated at 40° C, washed with a 1-percent solution of KNO₃ and titrated with alkali standardized by the use of acid potassium phthalate and the ratio 23 NaOH:1P.
^b Value obtained by standardizing the titrating solution with sodium oxalate through KMnO₄ and Na₂S₂O₈ and the 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 KIO₃ solution. Titer based on 93 percent of the theoretical factor.
^f Double dehydration with intervening filtration.
^g Diethylthiocarbamate-photometric method. See J. Research NBS 47, 380 (1951) RP2265.
^h H₂S-electrolytic method.

ⁱ Chromium separated from the bulk of the iron by hydrolytic precipitation with NaHCO₃ oxidized with persulfate, and titrated potentiometrically with ferrous ammonium sulfate.
^j Vanadium separated as in (i), oxidized with HNO₃ and titrated potentiometrically with ferrous ammonium sulfate.
^k Sulfuric acid digestion for 4 hr of a 0.5-g sample. See J. Research NBS 43, 201 (1949) RP202L.
^l Diphenylcarbazide-colorimetric method.
^m Semimicro distillation—Nessler photometric method. See Ind. Eng. Chem. Anal. Ed., 14, 137 (1942).
ⁿ Titrating solution standardized with a standard steel.
^o Weighed as ammonium hexaphosphomolybdate.
^p Copper-ammonia complex photometric method.
^q Photometric method.
^r Dimethylglyoxime precipitate titrated with cyanide.
^s Sulfur gases absorbed in H₂O₂ and H₂SO₄ titrated with standard NaOH using bromoresol-green indicator.

^t H₂S-CuO. Copper reprecipitated with Na₂S₂O₈ and titrated with KI-Na₂S₂O₃.
^u Sulfur gases absorbed in neutral H₂O₂ and titrated with sodium borate.
^v Silicomolybdate photometric method. See Anal. Chem., 21, 589 (1949).
^w Copper separated as the sulfide from a 10-g sample and determined by the diethylthiocarbamate-photometric method.
^x Vanadium precipitated with cupferron and determined by the phosphotungstovanadate-photometric method.
^y NaHCO₃ hydrolysis of a 10-g sample followed by mercury cathode separation. Vanadium titrated with 0.02N FeSO₄.
^z Sulfuric acid dehydration.
^{z1} Ether-cupferron separations on a 10-g sample. Vanadium titrated by the FeSO₄-(NH₄)₂S₂O₈-KMnO₄ method.

List of Analysts

1. Ferrous Laboratory, National Bureau of Standards, J. I. Shultz in charge. Analysis by E. June Maienthal, R. E. McIntyre, J. R. Spann, A. Skapars.
2. D. J. Hallisey, Jones and Laughlin Steel Corporation, Aliquippa Works, Aliquippa, Pa.
3. C. A. Trathowen, Jones and Laughlin Steel Corporation, Pittsburgh Works, Pittsburgh, Pa.
4. H. A. Patterson, United States Steel Corporation, South Works, Chicago, Ill.
5. O. W. Baldwin, United States Steel Corporation, Gary Steel Works, Gary, Ind.
6. R. W. Bley, Inland Steel Co., Indiana Harbor Works, East Chicago, Ind.
7. S. Partington, The Detroit Testing Laboratory, Inc., Detroit, Michigan.
8. J. B. Armstrong, Bethlehem Steel Co., Sparrows Point Plant, Sparrows Point, Md.

The steel for the preparation of this standard was furnished by the Jones and Laughlin Steel Corporation.

WASHINGTON, D. C., September 10, 1957.

A. V. ASTIN, Director.