U. S. DEPARTMENT OF COMMERCE

# National Bureau of Standards

## Certificate of Analyses

OF

## STANDARD SAMPLE 27 B—SIBLEY IRON ORE

[All results are based on a sample dried for one hour at 105°C]

| ANALYST | LABORATORY | TOTAL IRON | | | PHOSPHORUS | | SILICA[a] |
|---|---|---|---|---|---|---|---|
| | | Dichromate method | Permanganate method | Other methods | Gravimetric [a] | Volumetric [b] | |
| R. M. Fowler | National Bureau of Standards | 68. 21[d] | 68. 36[e]<br>68. 31[f] | 68. 25[g]<br>68. 19[h] | 0. 034 | | 1. 32[i] |
| C. B. Murray | Crowell & Murray, Inc., Cleveland, Ohio | | 68. 38[j] | | | 0. 036[k] | 1. 30[i] |
| R. M. Bundy | The Frank L. Crobaugh Co., Cleveland, Ohio | | 68. 22[m] | | | . 036[k] | 1. 27[i] |
| W. H. Bolger and E. J. Richards. | Robert W. Hunt Co., Chicago, Ill | | 68. 27[e] | | . 036 | . 035[n] | 1. 35[i] |
| B. O. Crites | Textor Chemical Laboratories, Cleveland, Ohio | | 68. 39[m] | | . 036[o] | | 1. 31[i] |
| A. M. Smoot | Ledoux & Co., New York City, N. Y | | 68. 37[e]<br>68. 38[f] | | | . 038 | 1. 31[i] |
| H. E. Slocum | Jones & Laughlin Steel Co., Pittsburgh, Pa | | 68. 34[f] | | . 035 | . 036 | 1. 31 |
| W. F. Lantz | Bethlehem Steel Co., Bethlehem, Pa | | 68. 36[j]<br>68. 40[e] | | | . 036[k] | 1. 29 |
| C. P. Larrabee | American Sheet & Tin Plate Co., Research Laboratory, Pittsburgh, Pa. | 68. 15[p] | | | | | |
| R. J. Ruff | American Steel & Wire Co., Donora Works, Donora, Pa | 68. 28[q] | | | . 039 | . 038 | 1. 30 |
| G. H. Halverson | American Steel & Wire Co., Duluth Works, Duluth, Minn | 68. 20[q] | | | | . 038 | 1. 29 |
| W. F. Muehlberg | American Steel & Wire Co., Newburgh Works, Cleveland, Ohio | | 68. 27[e] | | . 033 | . 037 | 1. 31 |
| Ruhe | Carnegie Steel Co., Carrie Furnaces, Rankin, Pa | 68. 25[q] | | | . 038 | . 039 | 1. 28 |
| V. Seyler | Carnegie Steel Co., Clairton Works, Clairton, Pa | 68. 19[q] | | | . 038 | . 039 | 1. 31 |
| D. Brown | Carnegie Steel Co., Duquesne Works, Duquesne, Pa | 68. 15[q]<br>68. 26[r] | | 68. 16[h] | . 036 | . 037 | 1. 29 |
| C. E. Nesbitt | Carnegie Steel Co., Edgar Thompson Works, Braddock, Pa | 68. 22[q] | | | | . 037 | 1. 28[e] |
| H. E. Beck | Carnegie Steel Co., Farrell Works, Farrell, Pa | 68. 28[s] | | | | . 038 | 1. 30 |
| C. D. Wright | Carnegie Steel Co., Mingo Works, Mingo Junction, Ohio | 68. 32[q] | | | . 039 | . 039 | 1. 31 |
| T. S. Woodward | Carnegie Steel Co., Ohio Works, Youngstown, Ohio | | 68. 30[e] | | . 036 | . 036 | 1. 30 |
| A. H. Ward | Columbia Steel Co., Pittsburg, Calif | | 68. 43[e] | | . 035 | . 036 | 1. 31 |
| L. A. Culberson | Columbia Steel Co., Provo, Utah | | 68. 44[e] | | | . 035 | 1. 31[i] |
| A. D. Beers | Illinois Steel Co., Gary Works, Gary, Ind | 68. 25[q] | 68. 27[e] | | . 036 | . 037 | 1. 32 |
| L. P. Chase | Illinois Steel Co., South Works, Chicago, Ill | | 68. 17[m]<br>68. 36[e] | | . 034 | . 037 | 1. 32[i] |
| F. W. Crouch | National Tube Co., National Works, McKeesport, Pa | 68. 18[q] | 68. 30[e] | | . 038 | . 037 | 1. 29 |
| S. S. Heide | Tennessee Coal, Iron & Railroad Co., Ensley Works, Ensley, Ala. | | 68. 43[e] | | . 036 | . 036 | 1. 29 |
| F. Fenwick | U. S. Steel Corporation, Research Laboratory, Kearny, N. J. | 68. 18[p] | | | | | |
| R. A. Barnes | Oliver Iron Mining Co., Coleraine, Minn | | 68. 35[e] | | . 035 | . 034 | 1. 28 |
| W. J. Trudgeon | Oliver Iron Mining Co., Ely, Minn | | 68. 38[e] | | . 036 | . 036 | 1. 31[i] |
| C. H. Webster | Oliver Iron Mining Co., Hibbing, Minn | | 68. 25[j] | | . 036 | . 036 | 1. 29 |
| W. L. Winn | Oliver Iron Mining Co., Ironwood, Mich | | 68. 30[e] | | . 036 | . 036 | 1. 30 |
| A. T. Gordon | Oliver Iron Mining Co., Virginia, Minn | | 68. 37[e] | | . 035 | . 036 | 1. 29[i] |
| Averages [1] | | 68. 22 | 68. 34 | 68. 20 | . 036 | . 037 | 1. 30 |
| Recommended values | | | 68. 23 | | | 0. 036 | 1. 31 |

[1] Values for constituents not established as accurately as the above values are: $Al_2O_3$=0.59, $TiO_2$=0.023, V=0.004, Cr=0.001, and Mn=0.03.

### 1934 INTERNATIONAL ATOMIC WEIGHTS USED IN ALL CALCULATIONS

(a) Weighed as $Mg_2P_2O_7$ after removal of the arsenic.

(b) Titrated with alkali standardized by the use of National Bureau of Standards acid potassium phthalate and the 23:1 ratio.

(c) Ore dissolved in HCl, residue fused with $Na_2CO_3$ and silica recovered by two evaporations with HCl. Ignited silica treated with HF and $H_2SO_4$.

(d) Reduced with $SnCl_2$ and treated with a slight excess of $HgCl_2$. Iron titrated potentiometrically under $CO_2$ with $K_2Cr_2O_7$ using weight burettes. The standard $K_2Cr_2O_7$ solution was prepared on the theoretical (weight) from the recrystallized and fused (in an electric salt.

Zimmermann-Reinhardt method. $KMnO_4$ standd against National Bureau of Standards sodium oxalate by McBride's procedure. Tests made at the National Bureau of Standards after the values *e* and *f* were obtained showed that the titre of a permanganate solution standardized with sodium oxalate by McBride's procedure is too high (see National Bureau of Standards Journal of Research **15**, 493 (1935) or RP843). This accounts in large part for the high results for iron that were obtained for *e* and *f*.

(f) Zinc reduction-$KMnO_4$ titration. $KMnO_4$ standardized by means of National Bureau of Standards standard sodium oxalate according to McBride's procedure. Iron value corrected for V and Ti present.

(g) Iron precipitated with cupferron after removal of silica. Ignited in oxygen and weighed as $Fe_2O_3$. Corrections were applied for V, Ti, and Si.

(h) $SnCl_2$ reduction. Excess removed with $HgCl_2$ and iron titrated in a $CO_2$ atmosphere with $Ce(SO_4)_2$ standardized against National Bureau of Standards standard sodium oxalate.

(i) Ore dissolved in HCl and silica dehydrated by baking at 110° C followed by solution, filtration, and a second dehydration. Ignited silica purified with HF and $H_2SO_4$.

(j) Zimmermann-Reinhardt procedure. $KMnO_4$ standardized against National Bureau of Standards Standard Sample Sibley Ore 27a.

(k) Titrating solution standardized by means of National Bureau of Standards Standard Sample Sibley Ore 27a.

(l) $H_2SO_4$ dehydration.

(m) Zimmermann-Reinhardt. $KMnO_4$ standardized against private ore standards.

(n) Alkali standardized by means of National Bureau of Standards standard benzoic acid.

(o) Weighed as ammonium phosphomolybdate.

(p) Potentiometric titration with $K_2Cr_2O_7$ as in (d), but in air.

(q) $SnCl_2$ reduction, excess $SnCl_2$ oxidized with $HgCl_2$ and iron titrated with a solution prepared from recrystallized $K_2Cr_2O_7$ either dried at 190 to 200° C or fused, using the theoretical factor and diphenylamine or diphenylamine sulfonate indicators.

(r) As in (q), but $K_2Cr_2O_7$ solution standardized on private ore standards.

(s) Solution and dehydration with $HClO_4$.

(t) Dichromate solution standardized against recrystallized $FeSO_4(NH_4)_2SO_4.6H_2O$.

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