

**METHODS FOR THE ANALYSIS OF
ORES, PIG IRON AND STEEL, IN
USE AT THE LABORATORIES OF
IRON AND STEEL WORKS IN THE
REGION ABOUT PITTSBURG, PA.**

Published @ 2017 Trieste Publishing Pty Ltd

ISBN 9780649503513

Methods for the Analysis of Ores, Pig Iron and Steel, in Use at the Laboratories of Iron and Steel Works in the Region about Pittsburg, PA. by Various

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VARIOUS

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over

METHODS FOR THE ANALYSIS

—OF—

Ores, Pig Iron and Steel

IN USE AT THE

Laboratories of Iron and Steel Works

—IN THE—

REGION ABOUT PITTSBURG, PA.

TOGETHER WITH AN APPENDIX CONTAINING VARIOUS
SPECIAL METHODS OF ANALYSIS OF ORES
AND FURNACE PRODUCTS.

CONTRIBUTED BY THE
CHEMISTS IN CHARGE, AND EDITED BY A COMMITTEE OF
THE CHEMICAL SECTION, ENGINEERS' SOCIETY
OF WESTERN PENNSYLVANIA.

EASTON, PA.
CHEMICAL PUBLISHING CO.
1893.

PREFACE.

These methods in use in the iron and steel laboratories of the region near Pittsburg, Pa., were collected and published by the Engineers Society of Western Pennsylvania during 1896. The supply of copies having been exhausted, in response to a continuous demand the publication in more convenient form has been undertaken by the Chemical Publishing Company who have been authorized to do so by resolution of the Society at a meeting held March 13, 1897.

These methods were detailed by those using them in response to the following circular sent out by the committee in charge:

CHEMICAL SECTION.
ENGINEERS' SOCIETY OF WESTERN PENNSYLVANIA,
PITTSBURG, PA.

In accordance with a resolution of the Chemical Section of the Engineers Society of Western Pennsylvania, the undersigned wish to ask your cooperation in an effort to collect, for publication in the Proceedings of the Society, the method of analysis in use in the various iron and steel works laboratories of the region.

In calling the attention of chemists to the plan and asking their aid in its fulfilment, it should be mentioned that it is the aim of the Section to secure accurate statements of analytical processes, describing with minuteness and clearness the successive steps, in order that the proposed compilation may represent as correctly as possible the present status of analytical chemistry as applied to iron and steel.

A full presentation of the methods in general use is likely to prove of interest and value, but the completeness and promptness of the responses received from a large number of chemists must determine the success of the measure.

In case you are willing to cooperate, you are requested to send to any one of the undersigned a full description of the methods you use for the determination of the following substances:

In Ores—Silica, iron, phosphorus, manganese.

In Pig Iron—Silicon, sulphur, phosphorus, manganese.

In Steel—Carbon (by combustion), sulphur, phosphorus, manganese, nickel.

SUGGESTIONS:

1. If the method is described in a text-book or journal, a mere reference

will suffice, but any deviations from the published methods should be noted.

2. In writing a description of a method it is very desirable that minute details should be given (*e. g.*, weights taken, volume of solution, temperatures, etc., etc.).

3. If more than one method is used, please describe the one in everyday use on which the commercial transactions of the firm are based.

4. It is earnestly requested that a reply be sent at the earliest possible date.

Each method will be published over the name of the sender. The work of the committee will be confined to collecting and arranging for publication without comment or discussion.

F. C. PHILLIPS,
Western University, Allegheny, Pa.

A. G. MCKENNA,
Duquesne Steel Works, Duquesne, Pa.

E. S. JOHNSON,
Park Bros. & Co., Pittsburg, Pa.

Committee.

The methods which have been received in response to this circular may be considered to represent the general practice of the chief iron and steel works in the region of Pittsburg and Western Pennsylvania.

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I. METHODS USED AT THE LABORATORY OF THE CARNEGIE STEEL CO., HOMESTEAD, PA.

By JOHN S. UNGER.

DETERMINATION OF SILICA IN ORES OF IRON AND MANGANESE.

To 1 gram of the ground ore in an 8-oz. beaker add 35 cc. of hydrochloric acid (sp. gr. 1.20), cover with a watch-glass, and boil gently on a hot plate for twenty minutes. Remove and rinse watch-glass and sides of beaker with 15 cc. of water, filter off insoluble matter on an 11-cm. filter, catching filtrate in an 8-oz. beaker, and wash with water until soluble matter is removed. The filtrate, which should not exceed 90 cc., is placed on a sand-bath and allowed to go to dryness. The filter is ignited and the residue fused with 8 grams of sodium carbonate. The fusion is run up around the sides of the crucible and then the crucible is cooled by dipping cautiously in an 8-oz. beaker containing 35 cc. water, finally turning the crucible on its side and leaving it. Cover beaker with a watch-glass and add hydrochloric acid gradually until effervescence ceases. After the fusion dissolves out of the crucible, remove with a glass rod and wash the crucible with water. The solution should not exceed 90 cc. Evaporate on a sand-bath to dryness. When both solutions are evaporated, moisten the residues with 10 cc. hydrochloric acid (sp. gr. 1.20) and leave on the bath for a few minutes; then add 20 cc. hot water, and filter through an 11-cm. filter, the first into a 16-oz. Erlenmeyer flask, washing out the beaker carefully, then filter the second through the same filter. If the alkaline salts have not all dissolved in the second add 20 cc. hot water and stir until dissolved; filter, and rinse the beaker with hot water until filter is perfectly free from soluble salts. Ignite the filter in a muffle, cool, and weigh as pure silica. The filtrate, which is preserved for further use, should not exceed 150 cc.

DETERMINATION OF IRON IN IRON ORES.

To the filtrate from silica (given under silica) add 12 grams

of Baker and Adamson's shot zinc and 10 cc. concentrated hydrochloric acid, and place in the neck of the flask a small funnel. Let the flask stand for about twenty or thirty minutes or until the iron is all reduced. Should the action on the zinc become feeble, add 10 cc. concentrated hydrochloric acid. When the iron is reduced test a drop of the solution with a drop of potassium thiocyanate on a white porcelain plate. If it shows no color or just a very faint pink, it is ready for titration. Have ready a 16-oz. wide beaker, a platinum triangle large enough to extend over the edges of the beaker, and a Gooch crucible with a tuft of glass wool $\frac{1}{4}$ inch thick at the bottom. Rinse the funnel into the flask with a wash-bottle and pour the solution through the Gooch crucible, receiving the solution in the beaker. Rinse the flask three times with about 10 or 15 cc. of water and wash the Gooch crucible once. To the filtrate in the beaker add 5 cc. concentrated hydrochloric acid and place on a hot plate for two minutes. The solution will now occupy 225 cc. Remove from the heat and titrate with standard bichromate solution, using 3 drops for each test toward the end, and adding the bichromate solution, 5 drops at a time, when almost done. Agitate each test drop by blowing gently, and continue the addition of the bichromate until the last test shows an absence of blue precipitate after agitating and standing thirty seconds. The bichromate used, multiplied by the factor, gives the metallic iron.

The potassium thiocyanate solution is made by dissolving 10 grams of the salt in 100 cc. water and is kept in a small bottle provided with a glass cap and a short piece of $\frac{1}{4}$ -inch glass tubing to be used as a pipette in taking out the test drops. The potassium ferricyanide solution is made by dissolving 1 gram of the salt in 100 cc. water and test drops are removed as with thiocyanate. The solution must be made up each day. One drop is used for a test.

The potassium bichromate is made by dissolving 52 grams in 12 liters of water, then shaking well and keeping the stock bottle protected from light.

The porcelain plate is 6 by 6 inches with 12 depressions 1 inch wide by $\frac{1}{4}$ inch deep on its surface.

The bichromate solution is standardized by dissolving 3 por-