

About five pounds of the air-dried plant were digested 24 hours with warm water and the brownish colored extract evaporated to a small bulk. The tannins were precipitated with basic lead acetate and the filtrate shaken out with chloroform in a separatory funnel. The chloroform extract was allowed to slowly evaporate and a gummy residue was obtained which, on standing, crystallized in a cell-like structure, crystallization beginning at a number of points and proceeding radially until intersecting. On melting, the mass recrystallized into a fibrous form. The crystals were colorless, had a very strong odor of coumarin and honey and melted at 65°. Ferric chloride gave no color to their water solution, showing the absence of vanillin. Fusion of the crystals with caustic potash gave acetic and salicylic acids.

The crystals of coumarin were purified by dissolving in ether and shaking out with 2% ammonia water in a separatory funnel. A small amount of a fragrant, low-melting, uncrystallizable substance was obtained on acidifying the ammoniacal extract and shaking out with chloroform. The ether solution, after the treatment with ammonia water, was allowed to slowly evaporate and the beautiful, characteristic rhombic crystals of coumarin were obtained having a melting point of 67°. By shaking out the water extract of the plant with ether according to Reinsch<sup>1</sup> a yield of 0.20% was obtained on the air-dried plant. Mature plants yield best and a large proportion of the coumarin exists in the leaf stalks. Among the several plants which have been found to contain coumarin<sup>2</sup>, *Hierochloa borealis* is the only Western species noted. This plant is found growing along streams of the Northwest.

C. E. BRADLEY.

Corvallis, Oregon.

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## REVIEW.

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### THE TESTING OF COPPER AND ITS BY-PRODUCTS IN AMERICAN REFINERIES.

BY GEORGE L. HEATH.

Received January 9, 1907.

A proper control of refining is attained by the determination of the quality of all supplies and fluxes, and by mechanical, chemical, and electrical tests of the metal from the ore, or concentrate, to the finished condition of commercial purity.

In an electrolytic plant, we have, also, working tests of vat solutions, slimes, blue vitriol, anodes, and bullion.

In the treatment of copper, your attention is called to a special factor, unimportant to the iron and steel metallurgist. I refer to the quality of high electrical conductivity, which makes the metal a necessity in the

<sup>1</sup>J. 1867, 439.

<sup>2</sup>Parry "Essential Oils," p. 354.

wonderful electrical development of the present and future. The maintenance of a high standard of quality, by elimination of all but slight traces of foreign impurities, is a problem of highest importance to both smelter and consumer. Less than 0.0020% of some elements markedly lower the electrical conductivity of copper.

In America, processes of analysis at Eastern refineries differ somewhat from those of the "Far West," because of the character of the material treated, so much of the converter pig and anodes of the West being shipped east for electrolytic refining. Lake Superior smelters have the simpler problem of reduction of ferruginous concentrates of native copper, containing no more than a trace of sulphur. Copper, produced from sulphide ores, is worked up from impure material to high grade by several steps. Any mistake in the series causes more of the original impurities to be retained, hence the necessity of "eternal vigilance."

The first logical division of tests is:—

1—Sampling and assaying of ores, mill concentrates and slags.

At Ore-smelting Works.

- A —Raw and roasted ores.
- B —Mill products and tailings.
- C —Rich slag from furnaces and converters.
- D —Waste slag and flue dust.

On Lake Superior.

- A —Mine rock with native metal.
- B —Concentrates and tailings.
- C —Reverberatory furnace slags.
- D —Cupola slag and flue dust.

#### SAMPLING.

1A—The vein rock of Lake Superior mines carries the native metal in particles so unevenly disseminated, that any valuation of a stope by any means, short of a mill test, is of little value. The sampling of ores is so well known to the profession, and has been so often discussed that little comment is advisable. Mechanical sampling machines, combined with intermediate roll crushing are the usual device for the preliminary reduction of large lots of ore. Some of the largest concerns find it best to adhere to the old method of hand-quartering or split-shovelling on an iron floor, for the final reduction from the quantity, leaving the machines to the amount sent to the assay office.

D. W. Brunton, Dr. S. A. Reed and others (*Col. School of Mines Quarterly*, vol. 3, No. 4, also vol. 6 (1885) p. 351), have given theoretical and elaborate rules for different classes of ores. Dr. Reed well stated that in order to get an average sample we must observe the following precautions:—1st—Adequate mixing, 2d—Impartial selection, 3rd—Proper relative comminution. Mill concentrates, being fine, are easily sampled by hand as material is running from chutes, or by rejecting a fixed proportion when shovelling. Concentrates of native metal should be sampled while still wet, so that the particles of lighter rock may adhere to the heavy copper. The material is preserved in tight cans and the water estimated.

1C—At some places, pieces are broken from cakes of rich slags after cooling. Wherever it can be done, a better practice in my experience, is to take small buttons with a test ladle from the flowing stream after it leaves the settler. With rich slags of native copper, metallic prills have to be picked from the sieves during the grinding, and separately weighed.

1D—Waste slags are handled similarly, although a frequent practice consists in dipping an iron rod into the molten slag and knocking off the

coating. This does not always show the right proportion of prills of matte or metal in the slag stream.

## ASSAYING.

*I A & B*—Pulverized ores and mattes are generally assayed for copper by titration of the solution with potassium cyanide, (*Peter's Modern Copper Smelting—Low's Technical Methods of Ore Analysis, 2d Edition*), potassium iodide, (*This Journal*, **24**, 1082, (1902)), or potassium permanganate. (*Ibid.*, **20**, 610, (1898); *Ibid.*, **24**, 580, (1902)), after precipitation of copper as sulphocyanate. A. H. Low, of Denver, has probably done the most to perfect these titration processes, and his text-book gives the latest modifications.

George A. Guess (*Trans. A. I. M. E.*, **36**, 605, (1906)), has recently developed a rapid electrolytic method for both copper and lead in ores. He employs a mixture which will prevent the co-precipitation of arsenic or antimony, when a little is added to each assay. It is prepared by boiling No. 4 hard oil (St'd. Oil Co.) with strong nitric acid, and then filtering out the grease, which is rejected. Mr. Guess claims that very strong currents may be used. For Lake Superior metal, slags, or tailings the electrolytic (*Eng. and Min. J.*, April 20, 1895, p. 369,387) method is used for accurate work, and generally without any filtration from the sand or insoluble residue.

Mill concentrates of native copper (*This Journal*, **24**, 699, (1902)) are usually tested by subjecting 1000 grain samples to the Lake Superior Fire Assay. The samples which have been preserved in tight cans are mixed upon a tray, about 14x19 inches in size, made by cutting out one side from a hard rubber photo-developing tray. Details have been published elsewhere.

*ID*—The copper in waste slags from ore, or remelting cupolas, can be estimated by titration, or battery, but is more frequently controlled by a colorimetric assay, the "Blue Test," accounts of which have been printed by the author. (*This Journal*, **19**, **24**, (1897)).

The work of Mr. Thorn Smith, (*Eng. and Min. J.*, **75**, 295), on the analysis of slags, has shown causes of marked variations between chemists, and the precautions necessary with material which frequently runs very high in oxides of iron and alumina.

Blue color standards, prepared with copper as sulphate, dissolved in ammonia, (1 vol.  $\text{NH}_4\text{OH}$ , 0.9 sp. gr. to 5 vols. of water), and preserved in sealed, glass-stoppered, cylindrical bottles, will keep a year, without change.

*Gold and Silver in Copper Ores.*—Success in this assay is mainly a matter of good judgment in fluxing and firing, so that the copper shall be carried with iron, etc., into the slag, and details are found in the text-books of Furman, Aaron and Brown.

The writer has found that a large excess of litharge is desirable in the direct firing of copper ores, and that good results may be obtained by changing all copper present to sulphide by Aarons' formula, adding nails and an excess of sulphur and soda according to the recommendation of Prof. R. W. Lodge.

Some assayers withdraw nails ten minutes before pouring, and at least twenty minutes at quiet fusion, I think, should be given, to allow the copper to pass into the slag as sulphide. (*Aaron's Assaying Part I*).

The complete analysis of slags is carried out by regular gravimetric methods. Precipitates of iron and lime are redissolved and titrated, as also lead or zinc, when in sufficient quantity.

Lead, alone, is weighed as sulphate, or titrated by ammonium molybdate, and zinc, or nickel, by potassium ferrocyanide. (*Mining World*, 544, 1906).

The Western Association of Chemists and Metallurgists is attempting to adopt uniform methods for the analysis of ores and supplies. (*Chem. Eng.*, 25, 287, (Sept., 1905); also, *This Journal*, 28, R. 184, (1906)).

Copper ores, or native copper, too rich to test for gold and silver by direct fusion, are treated with nitric acid, recovering the precious metals as described later, under the head of refined copper.

#### SECOND DIVISION:—METALLIC COPPER.

Unusual accuracy is necessary in the analysis of the metal, not only on account of the precious metals in such material as Western converter copper, but on account of the marked effect of impurities on the refined product, as already noted, and the fact that these impurities segregate on cooling from a liquid to a solid state.

*Sampling of Metal.*—The technical analysis of the metallic product, in the United States, is confined to a limited number, but it is becoming every year more important, as requirements become more severe, that none but delicate, and strictly accurate methods should be employed for high grade material.

Edward Keller's paper (*Trans. A. I. M. E.*, 27, 106 (1897)) thoroughly discussed the proper sampling of metal for gold and silver, and in accordance with his results, some works do not drill the cast pigs, or anodes, but cast a small thin plate with a hand ladle from the molten charge, just before dipping or pouring. These plates are made  $\frac{1}{2} \times 6 \times 6$  inches, or for large furnace charges of rich material,  $1 \times 12 \times 12$  inches in size. A set of holes is drilled through the plate, keeping away from the edges a distance of at least twice the thickness of the casting, to avoid all variations due to segregation from the edges inward.

Commercial reasons make it necessary to sample at Eastern works and custom laboratories an immense amount of Western metal in the form of pigs, slabs, and anodes. For this purpose, holes are drilled in a specified number of castings from each carload, the number and position of the holes being fixed at each plant to suit their requirements. Bars are sometimes drilled in sets of five from top and bottom. Certain works use templet plates, perforated with rows of numbered holes, and of sizes to slip over any casting received. By this means, one hole can be drilled in a fixed order of progression, in each and every casting.

When refined cakes, wire-bar, and ingots are to be tested for purity, the surface oxides should be removed before drilling, and all drillings should be carefully inspected for dirt, and tested with a magnet for iron.

From my experience the best sample of an ingot, or bar, can be obtained by drilling holes one inch deep with a  $\frac{3}{8}$ - or  $\frac{1}{2}$ -inch drill, taking three holes diagonally across the top, three horizontally on one side, and three diagonally across the bottom, in reverse order to the upper set. The mixed borings constitute the sample.

#### ANALYSIS.

The following tests are made at refineries and foundries on crude and refined metal.

## 1.—Chemical.

## 2.—Mechanical.

## 3.—Electrical.

In addition to these, the science of metallography or micro-photography will be, in future, a very valuable adjunct to the older means of investigation, as it has already become to the iron and steel industry. The methods of polishing, with some modifications to suit a softer metal, are practically the same as for iron, details of which are generally known, and may be referred to, if desired, in vols. 5 and 6 of the *Metallographist*, or Prof. H. M. Howe's *Metallurgical Laboratory Notes*.

The writer has described the mechanical and electrical testing of the metal in a previous paper, published by the Lake Superior Mining Institute. (*Proceedings of the Lake Superior Mining Institute*, 7, 68-82, (1901)).

*1.—Chemical Analysis.—Gold and Silver.*—The combination wet method and fire assay have been compared in a paper presented by Dr. Ledoux to the A. I. M. E. in 1894. (*Trans. A. I. M. E.*, 1894, 250).

A later account of the methods, employed at the Mint, is given by Whitehead and Ulke. (*Eng. and Min. J.*, 1898, pp. 189, 233, 250). The process of Godshall, (*Eng. and Min. J.*, 228, 470, (1900)); treats the copper solution with a very little hydrogen sulphide to carry down the last trace of gold, and has been variously reported on by those who have tried it. In the hands of the writer, it has given no better results than the regular method.

The direct fire assay I carry out as follows:—Ten samples of 0.1 assay ton are scorified, each with 50 grams of test lead, combined in pairs, the five rescorified with additional lead until soft enough to cupel. Silver buttons are weighed and parted together. Although much experimenting has been done, with wet methods, the direct "all-fire" assay still seems, in most hands, to give a little higher result on gold.

If the metal is quite free from silica and sulphur, (*Trans. A. I. M. E.*, 31, 484, (1901)), much time may be saved, and one chance of loss eliminated by cupelling the gold and the silver chloride direct without scorification, according to detailed directions published elsewhere. Such a method is in use in two or three refineries.

*Determination of Metallic Copper.*—The electrolytic assay (*Trans. A. I. M. E.*, 27, 390 and 962, (1897)) is nearly always employed, and a former paper gives the particulars to which the reader is referred. 4 to 5 grams is the least quantity of sample for assay, which will allow the chemist to maintain his work within an ordinary limit of error of 0.01%.

The writer now uses the following rapid method:

(A). Dissolve 5 g. drillings, (freed from iron by a magnet), at 80-90° Centigrade, in a mixture of 7 cc. C. P. nitric acid, (sp. gr. 1.42), 10 cc. sulphuric acid (sp. gr. 1.84) and 25 cc. water. In practice, a bottle of acid mixture is made and 41 cc. taken for each assay. Allow to stand till most of the red fumes are evolved, dilute to 120 cc.; introduce weighed electrodes, and electrolyze with current  $N.D_{100} = 0.75$  amp., (counting both sides of cathode) until colorless, then reduce to 0.5 ampere. Finally test the solution for copper, as given in the following paragraph.

The cathode is a sheet 5 cm. high and 10 cm. wide bent into cylindrical shape, and provided with a stem of heavy wire 6.5 cm. above the plate. Time of assay 20-24 hours.

(B). W. H. Bassett dissolves the 5 g. sample in a mixture of 12 cc. of

dilute sulphuric acid, (1 : 1 by vol.), 3 cc. strong nitric acid, (1.42 gr.) and 20 cc. of water, and then adds, after dilution, 10 cc. of a 20% solution of C. P. sodium sulphite, free from chlorine. The other conditions are the same as in *A*.

Copper may be deposited by the current, in presence of arsenic, antimony, bismuth, selenium, or tellurium, by methods (*This Journal*, 26, 1120, (1904)), devised by the writer; and according to the statement of Mr. Guess, by the addition of a peculiar oily nitro-compound, invented by him.

In brief:—Selenium and tellurium are previously removed by treatment of the sulphuric acid solution with a sulphite, or sulphurous acid gas. Antimony and arsenic can be separated by the addition of 3 cm. of a solution of ferric nitrate, (1 cc. = 0.01 gram of iron), and precipitation with an excess of ammonia. The precipitate is purified again, dissolved the metals brought down as sulphides, the antimony extracted by a trace of sodium sulphide, and any trace of copper redissolved and returned to the main solution.

It was also discovered that copper will deposit pure from a slightly acid solution, nearly saturated with ammonium nitrate in presence of an excess of arsenic and 0.005% of antimony. The details of the method have been recently improved, so that the changes will be noted here:—

Prepare a nearly saturated solution of ammonium nitrate free from chlorine by carefully adding strong ammonia to concentrated C. P. nitric acid.

To the solution of about 5 grams of copper, in 20 cc. of nitric acid (sp. gr. 1.42), add 50 cc. of the stock solution and 1 cc. of strong sulphuric acid. Neutralize the solution with ammonia until the last drop produces a permanent precipitate. Redissolve this by 2 cc. of strong sulphuric acid. The proper acidity of the liquid is the important point, and when once determined by a few experiments, should be adhered to exactly.

Fill the beaker with the ammonium nitrate solution to the desired volume and electrolyze. The test for complete precipitation is made by transferring 1 cc. of the liquid, when colorless, to a porcelain tile and adding a few drops of hydrogen sulphide water. A trace of copper will show at once: arsenic a little later.

The beaker which is 2¼ inches in diameter and about 5 inches high, is covered with split watch glasses, which are washed down as soon as the liquid becomes colorless. The solution is finally siphoned out while a stream of water is poured in.

Current = N. D.  $_{100} = 0.75$  ampere, (counting both sides of the cathode) until the solution is almost colorless, when the strength should be reduced to 0.5 ampere to prevent oxidation, and the beaker washed with a jet. N. D.  $_{100} =$  the density in amperes per square centimeter of cathode surface.

The ammonium nitrate solution can be regenerated by treatment with hydrogen sulphide, or by addition of excess of ferric nitrate and excess of ammonia. Several chemists are using this scheme, and report good results.

*Oxygen in Copper*.—To the German chemist, Hampe, (*Z. Berg. Hütten und Salinen-Wesen*, 1873; also *Archbutt, Analyst*, 25, 253, (1900); *Hofman, Trans. A. I. M. E.*, 34, 671, (1904)), is due the only method that

combines scientific exactness with adaptability to technical work. He determines the loss of weight of clean copper filings by heating to a constant weight in a current of perfectly pure hydrogen gas, the metal being contained in a weighed bulb of hard glass. For fuller data refer also to an earlier paper by the writer. (*This Journal*, **27**, 313 (1905)).

In addition to the solutions specified for the purification of the gas, it is recommended to insert, after the second tube, a bottle of a strong solution of potassium permanganate.

The copper is cleaned with petroleum ether, or according to Archbutt with ethyl ether, and any evolved sulphur is absorbed by alkaline cadmium chloride solution, and its weight allowed for.

When all other elements have been determined in a refined copper, and their sum added to the copper and the total deducted from 100%, the difference may be assumed to be the oxygen present, without any sensible error.

*Sulphur*.—The electrolytic method (*This Journal*, **17**, 714, (1895)) of the writer is in common use. The copper is removed by electrolysis of the  $\text{HNO}_3$  solution and the filtrate concentrated with sufficient soda to hold the sulphur.

*Metallic Impurities in Copper*.—Special methods for each element, or similar group, are the general rule in technical laboratories, when a complete analysis is not required. (*J. Soc. Chem. Ind.*, 421, (1894)). For a complete analysis, the scheme adopted is some modification of the original method of Dr. Hampe.

For the determination of gold, silver, selenium, tellurium, arsenic, antimony, tin and bismuth, the writer dissolves 50 grams of metal in 200 cc. of nitric acid, (sp. gr. = 1.42) and effects the preliminary separation of the foreign elements from the bulk of the copper in one of three ways to be given in the order preferred.—

A—By precipitation with excess of ferric sulphate and ammonia.

B—By throwing down the copper as sulphocyanate by a calculated quantity of potassium sulphocyanate, after charging the solution with sulphurous acid at a temperature of 40 degrees centigrade.

C—By electrolytic deposition of 98% of the metal (or 90% in the case of crude copper), in presence of nitric and sulphuric acids, and 2.5 grams of ammonium nitrate, as directed by Mr. Heberlein. (*Trans. A. I. M. E.*, **27**, 962, (1897)). 50 grams of metal require for solution about 60 cc. of nitric acid (sp. gr. 1.42), 100 cc. of strong sulphuric acid and 400 cc. of water. The liquid is diluted with an equal volume of water before electrolysis. The solution is, when deprived of the copper, evaporated until all nitrates are driven off, the residue dissolved in dilute hydrochloric acid, and metals removed by repeated treatment with hydrogen sulphide gas.

The arsenic group are extracted by a little hot, dilute sodium sulphide, and the extract acidified with hydrochloric acid, the sulphides filtered on asbestos felt, and redissolved by the same acid. (2:1 of water), with the addition of a little potassium chlorate. Strong acid is then added, until there are 2 parts of acid to 1 part of water in the whole solution.

Arsenic is precipitated by hydrogen sulphide from the warm solution, and antimony after diluting to four times the original volume.

Method A was devised by chemists at the Baltimore Copper Works.

The writer employs ferrous sulphate, and oxidizes it by nitric acid, taking 20 times as much ferric oxide as the weight of arsenic or selenium and tellurium present. The nitric acid solution of the sample is treated with the iron salt, heated to boiling, and ferric hydroxide precipitated by ammonia, settled ten minutes, and filtered through a 15 cm. filter. The filtrate is acidified, 0.5 gram ferrous sulphate added, and excess of ammonia to ensure the removal of the last traces of arsenic.

The arsenic, etc., is removed from the ferric hydroxide as in method C, or the sodium sulphide extract of arsenic and antimony may be acidified with sulphuric acid, instead of hydrochloric, the precipitate after settling, filtered through asbestos felt under suction, and dissolved in red, fuming nitric acid. Dilute with an equal bulk of water, filter out the asbestos, and evaporate to dryness with enough sodium carbonate to unite with the arsenic and antimony. Add 0.5 cc. hydrochloric acid, 0.1 gram of solid tartaric acid and 5 cc. of water, warm and filter on a 2 cm. paper. Make slightly alkaline with strong ammonia, and dilute to 10 cc. Add 2.5 to 4 cc. of magnesia mixture, Fresenius' formula, and dilute to 20 cc. with the strongest ammonia water, stir five minutes, and allow to settle over night. Transfer the ppt. with the aid of the filtrate to a 3 cm. filter, and wash with about 5 cc. of dilute ammonia (1:3), from a wash bottle. Moisten the dried filter, etc., with acid ammonium nitrate, and ignite.

If the arsenic is over 0.1%, increase the precipitant, and the final volume of the solution, one-half. The filtrate is always to be tested with a little more magnesia mixture, and the antimony and tin are thrown down from the acidified filtrate, and separated from each other by F. W. Clarke's method of precipitation in a boiling saturated oxalic acid solution by hydrogen sulphide. (Total volume of liquid not over 40 cc.).

*Method D*—A few chemists prefer to distill off the arsenic, and sometimes antimony, as follows:—

1.—By the Fischer, or Lundin, method with excess of ferrous sulphate and hydrochloric acid, saturated with the hydrochloric acid gas. (*Classen, Analysis by Electrolysis; Ulke, Eng. and Min. J.*, 18, 727, (1899)). With proper precautions, this gives accurate results.

2.—By the method of Aller, who reserves  $\frac{2}{3}$  of the drillings to reduce the hydrochloric acid solution of the part already dissolved.

The residue is then evaporated almost to fusion, and distilled with 75 cc. of a saturated solution of zinc chloride to remove the antimony. The writer has no experience with this scheme.

*Method E*.—A good alternative device for the final precipitation of arsenic, (after separation of the antimony and tin), consists in the treatment of the nitric acid solution, from which most of the acid has been evaporated, with a slight excess of an emulsion of zinc oxide, to render the liquid strictly neutral. (*Furman's Manual of Assaying, last ed.—Chapter on Analysis of Copper* ).

Add a slight excess of silver nitrate, and stir the cold liquid five minutes, settle, filter, wash out all silver nitrate, redissolve the arsenate of silver in dilute nitric acid, and finally precipitate and weigh it as chloride.

25 parts of arsenic correspond to 107.93 of silver.

The arsenic, if less than 0.01%, may be oxidized to arsenic acid by grinding and fusing an aliquot, weighed portion of the gently ignited iron oxide precipitate (of Method A) with 8 parts of an equal mixture of sodium carbonate and potassium nitrate.



The direct weighing of arsenious sulphide, often practiced, is not to be recommended, for it is apt to retain free sulphur, in spite of every precaution.

*Lead, Zinc, Cobalt, Nickel, and Manganese.*—40 to 50 grams of copper drillings, carefully tested with a magnet, are dissolved in a mixture of acids, as outlined in method C for arsenic, and the copper completely removed by electrolysis. The small platinum anode sheet is removed and replaced by another when the copper is about half deposited, and both anodes are quickly washed, dried and weighed to determine the lead or manganese deposited. An additional trace of this element may be obtained later on, after the copper-free liquid has been evaporated to dryness in a porcelain casserole and the sulphuric acid driven off. Time of precipitation about 20 hours, with a revolving anode (at 600 to 1000 r. p. m.), less than 9 hours.

The reader is requested to refer to an earlier paper for more complete data. (*This Journal*, 27, 317, (1905)). The residue from evaporation is redissolved in a few cc. of dilute sulphuric acid and any lead sulphate filtered off. Charge the liquid with hydrogen sulphide gas, filter and test precipitate for a trace of lead. The filtrate is oxidized, and the iron precipitated twice by ammonia. Test filtrate for manganese, if its presence is suspected, otherwise acidify the filtrate slightly with acetic (or formic acid), add one-fifth its volume of glacial acetic acid, (or formic acid), and throw down the zinc in the cold, as sulphide.

The solution is then neutralized, the cobalt and nickel deposited as sulphides, and the cobalt separated, if desired, by potassium nitrite. The nickel and cobalt are generally deposited as metals, from the ammoniacal solution of their sulphates, using as anodes, old soft platinum wires, which are not attacked so quickly, by the ammonia, it is claimed, as those of smooth, hard metal.

Manganese will not wholly deposit on the anode during the electrolysis of the copper, as has been proved by several chemists.

*Bismuth.*—This element is usually determined in a separate 50 g. sample by repeated precipitation with excess of ammonia, and ammonium carbonate, in presence of a very little ferric hydroxide. It may finally be freed from the last trace of copper by precipitation as sulphide, in presence of an excess of potassium cyanide. It is weighed as oxide, or estimated by the color test of F. B. Stone. (*J. Soc. Chem. Ind.*, 6, 416; *J. Anal. Chem.*, 401, (1887)),

*Selenium and Tellurium.*—Although these elements may be separated with, or before, the arsenic and antimony in carrying out the methods for the latter, it is the usual practice to make a special test by the method of Edward Keller, (*This Journal*, 22, 242, (1900)), or separate them by charging the sulphuric acid solution of the copper with sulphurous acid gas, as in method B for arsenic.

Mr. Keller throws out the selenium from the copper solution by adding a large excess of an iron salt, and precipitating the ferric hydroxide with ammonia, as in method A for arsenic.

The solution of the sulphides of selenium and tellurium is evaporated to dryness on the water-bath with an excess of nitric acid. The residue is boiled with 200 cc. of hydrochloric acid to remove traces of nitric acid, and the selenium thrown down with sulphur dioxide gas, in strong solution, and the tellurium from the diluted fluid.

If a separation by this means is not desired the two elements may be reduced by stannous chloride in dilute hydrochloric acid solution or by hydroxyl amine, as recommended by Dr. H. F. Kellar. (*J. Franklin Inst.*, July, 1894); also *This Journal*, 16, 785. (1894).

*Fat Solutions and Slimes*.—These materials are usually controlled by special working tests, of which Titus Ulke has given a complete account. (*Ulke, Mineral Industry*, 2, 276. (1893)).

*Electrical Resistance*.—Refineries make tests of transverse strength, etc., on Riehle machines, but the most important physical test on the metal is the determination of its electrical conductivity. Many years ago, copper wire was tested in 20 or 30 foot lengths, on ordinary Wheatstone Bridges. The modern technical method involves the use of short lengths of wire, which are measured on low-resistance instruments, specially designed so that all parts shall be at the same uniform temperature, and all resistances of lead wires, plugs, or contacts are allowed for or eliminated.

Not only should such instruments be compared with Government standards, but a carefully tested set of copper wires should be provided, so that the instrument may be checked from time to time and any alteration detected. After handling wire samples they should remain on the Bridge at least ten minutes before testing. A distance of a foot from the body of the observer, will frequently make a difference of 2° F., equivalent to 0.4 per cent. change in electrical resistance.

Notwithstanding the number of supposedly good bridges on the market, there is room for improvement, as instruments from the same maker sometimes show unexplained differences when checked on the same samples of copper wire, and there appears to be a slight change in resistance of soldered joints, which one maker stated, is due to a very slow crystallization of the metal in the solder.

The foregoing methods of testing and chemical analysis to which frequent references have been made, are those in most common use, which the writer has found to give the careful observer an accurate knowledge of copper, and its by-products.

## NEW BOOKS.

INORGANIC QUALITATIVE CHEMICAL ANALYSIS FOR ADVANCED SCHOOLS AND COLLEGES BY WILLIAM STOWELL LEAVENWORTH, M. SC., Professor of Chemistry, Olivet College, 1906. The Chemical Publishing Co., Easton, Pa. London: Williams and Norgate. pp. VI + 153. Price, \$1.50.

This manual plainly reflects the experience of a painstaking teacher, and fulfils its purpose "to occupy an intermediate position between the elaborate treatise and the skeleton outline." The book opens with some introductory advice on the general operations of analysis. This is followed by a series of experiments with separate solutions of the salts of the metals which constitute each qualitative group, these experiments being illustrative of those variations in behavior and properties of the individual metals upon which their separation and detection depend. From the notes of these experiments the student is expected to devise a workable method of separation. In Part II the important reactions of