

d. Amounts of the Different Forms of Proteids in Cottonseed.—As already shown, the proteid matter of cottonseed soluble in water, consists almost wholly of proteose. Making full allowance for incomplete extraction and loss, this does not exceed 0.75 per cent. of the oil-free meal. The highest yield of globulin recovered in the preceding extractions was 15.83 per cent. of the oil-free meal, and contained 42.3 per cent. of the total nitrogen. After repeated extraction with potash water, the residue contained, in the case where extraction was most complete, 11.4 per cent. of the total nitrogen, showing that 88.6 per cent. of the total nitrogen had been dissolved. The difference between the percentage of nitrogen removed by sodium chloride solutions and that extracted by weak potash, represents the proteid dissolved by potash water which is not soluble in saline solutions, and which corresponds to 46.3 per cent. of the total nitrogen, assuming all this nitrogen to be present in proteid form.

These data may be tabulated as follows:

	Air-dry and oil- free meal.	Per cent. of Total nitrogen.
Proteose.....	0.75	2.0
Salt-soluble proteid. Edestin.....	15.83	42.3
Alkali-soluble and salt-insoluble proteid.	44.3
Proteid insoluble both in salt and alkali.	11.4
		100.0

ON THE ANALYSIS OF AMERICAN REFINED COPPER.¹

BY HARRY F. KELLER.

IT is generally conceded that the presence even of minute quantities of other elements in metallic copper has a marked influence upon its physical properties, and especially upon its electric conductivity.

The copper produced in the Lake Superior region is relatively free from injurious admixtures, and is, therefore, generally preferred when a high conductivity is desired. "Lake" copper is always quoted a trifle higher than the metal from other localities.

Of late years the production of copper from impure sulphide ores has grown enormously, and, at the same time, the quality of the product has steadily improved. It has, indeed, become

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possible through the introduction of the Manhés converter and the electrolytic process of refining, to obtain from matte a metal nearly, if not quite, as pure as the native copper of Lake Superior. Unfortunately, it appears that the very slight traces of the impurities retained in it affect the conductivity to a greater extent than is the case in the best lake copper. Silver and iron are the chief impurities of the latter, while electrolytic copper not infrequently contains small quantities of bismuth, antimony, arsenic, selenium, and tellurium.

That the exact manner in which these elements affect the physical properties of copper is not better understood, is, undoubtedly, owing to the lack of rapid, and at the same time accurate, methods of ascertaining their relative amounts. It is astonishing to note that the works on quantitative analysis, published in the English language, entirely neglect copper analysis. To my knowledge the important contributions to this subject by Hampe, Fresenius and other German chemists have not been translated.

For several years past I have given considerable attention to this subject. My analyses include the metal from the Lake mines, as well as copper obtained from Western mattes.

To the former, I found Hampe's original method¹ of analysis well adapted. This consists essentially in precipitating, by electrolysis, the greater part of the copper, and separating the metallic impurities in the usual manner from the remaining solution. The non-metallic elements such as oxygen and sulphur, are determined in separate portions of the sample.

For obvious reasons it is not desirable to employ this method in analyzing electrolytically refined copper. Accurate results can be obtained by following the plan proposed by Fresenius,² but the separation of large quantities of copper by means of hydrogen sulphide, and the difficulty of accurately weighing bulky solutions, render this method very objectionable. A far more convenient process is the one recently published by Professor Hampe.³ It is also more rapid than either of the two methods mentioned, and, with some modifications, it is especially

¹ *Ztschr. f. Berg. Hütten u. Salinenwesen*, 21, 218, and 22, 93.

² *Ztschr. anal. chem.*, 21, 229.

³ *Chem. Ztg.*, 1893, 92, 1691.

well adapted for the analysis of most of the American refined coppers.

The process depends upon the removal of most of the copper as cuprous sulphocyanate.

The manner in which I practice it is briefly as follows:

Twenty-five grams of material are placed in a tall, lipless beaker and treated with a mixture of 200 cc. of water, forty-five to forty-six cc. of nitric acid (sp. gr. 1.21), and twenty-five cc. of concentrated sulphuric acid (in case that much antimony or bismuth is present, as much as 100 cc. of sulphuric acid may be taken). The beaker is covered and heat applied until nitrous fumes are no longer given off and the solution then diluted with 200 cc. of water to prevent the separation of copper sulphate. A rapid current of sulphur dioxide is now conducted into the liquid, the temperature of which is maintained at about 40° C. When the nitric acid is destroyed and the red fumes have disappeared, the liquid becomes turbid, owing to the precipitation of silver, selenium, and tellurium. To insure the complete separation of the silver, a drop or two of hydrochloric acid may be added. After standing for twenty-four hours in a warm place, the solution is poured through a small filter into a graduated flask of two liters capacity. It may contain bismuth, antimony, arsenic, iron, nickel, and cobalt.

The sediment retained by the filter is composed of gold, silver, silver chloride, selenium, tellurium, possibly also lead sulphate, and traces of bismuth and antimony.

To determine the elements in the filtrate, the greater part of the copper must first be removed. To this end a measured amount of standard potassium sulphocyanate solution (one cc. = 0.05 Cu) is gradually added, whilst a current of sulphur dioxide is passed into the liquid. An excess of the sulphocyanate should carefully be avoided; it is preferable to leave some of the copper in the solution which should now emit a perceptible odor of sulphur dioxide. The delivery-tube is withdrawn and rinsed off into the flask, the contents of which are then made up to the mark. To effect a thorough mixing it is well to pour the contents into a dry beaker, and to retransfer them several times from one vessel to the other. The precipitate is allowed to set-

tle and an aliquot portion, say 1,800 cc. of the solution, filtered off. The separation and estimation of the different metals is effected according to the usual analytical methods. It is hardly necessary to state that the sulphur dioxide must be expelled before hydrogen sulphide is passed into the liquid.

In calculating the amounts of the metals contained in the entire liquid, we must allow for the volume occupied by the precipitate. According to Hampe, the specific gravity of the sulphocyanate is nearly three, and since twenty-five grams of copper would give about forty-eight grams of this salt, the volume of the latter would be sixteen cc. The actual volume of the solution, therefore, is $2,000 - 16 = 1,984$ cc. Suppose *e. g.*, we had filtered off 1,800 cc., and found in it 0.020 gram of arsenic, then

$$\frac{1984 \times 0.02}{1800}$$

would be the amount of arsenic in the sample taken.

There yet remains to describe the analysis of the insoluble portion from which the main solution was filtered. It may contain, as I have already said, gold, silver (both in the metallic state and as chloride), selenium, tellurium, lead sulphate, and traces of bismuth and antimony, occasionally also copper.

It is best to detach, as completely as possible, the dry mixture from the filter, and to destroy the latter with fuming nitric acid in a small porcelain casserole. The detached portion is then added, and treated with the nitric acid till completely oxidized. After evaporation on the water-bath of the free nitric acid, the residue is digested with dilute hydrochloric acid, which leaves the silver in the form of chloride. This is filtered off, purified, and determined in the usual way. From the filtrate, selenium and tellurium may be thrown down by means of hydroxylamine hydrochloride. In the cold this reagent seems to have little effect, but upon warming, a reddish turbidity is observed which gradually collects in the form of dark flakes. These represent all the selenium and tellurium; they are collected on a tared filter and dried at 105° – 110° C. to constant weight.

I have not been able to effect a clean separation of the two elements. Approximate estimations were made by fusing the

mixture in a current of hydrogen with potassium cyanide; the aqueous solution of the fused mass was then exposed to the air, which causes most of the tellurium to separate.

Gold, lead, and the traces of antimony and bismuth are determined by the ordinary methods.

NEW BOOKS.

SELECT METHODS IN CHEMICAL ANALYSIS, CHIEFLY INORGANIC, BY WILLIAM CROOKES, F. R. S., ETC., ETC.; EDITOR OF "THE CHEMICAL NEWS." Third edition, rewritten and enlarged; illustrated with sixty-seven wood cuts. London, and New York; Longmans. 1894. Price \$3.

This book is without padding and is but a bare, yet voluminous, statement of analytical fact. It is sufficiently explanatory to meet the wants of the average worker. It is minute in important details. It gives concise and clear, yet full, directions for separation and determination of all known elements, and has something to say of the hypothetical ones. And while but little is stated concerning those processes well understood by analysts, there is set forth about all which concerns the newer and verified ones. The book contains a skillful digest of the best of analytical literature down to the middle of the present year.

After the preface, come thirteen pages of index to captions. The next forty-two pages are given to potassium, sodium, lithium, cesium, rubidium, and ammonium, stating several methods for determination of each of them when found in various combinations. Chapter II relates to barium, strontium, calcium, and magnesium. The third chapter gives sixty-eight pages of the masterly digest of the literature pertaining to cerium, lanthanum, didymium, samarium, thorium, glucinum, the yttrium metals, titanium and zirconium. In all, there are 697 octavo pages of analytical methods and processes, followed by a copious index to subjects and to authors' names. There is little risk in stating that the advanced worker will here find, at least, something he did not before know regarding his own immediate field. The volume is invaluable.

The author has something to say to him who is constrained to use chemistry as the carpenter does his rule, and the least