

improbable that, especially with susceptible individuals, those oysters containing the larger quantities of copper might be a cause of illness.

In respect to the mode of analysis, it may be of interest to state that in most cases the oysters were digested with a minimum of sulphuric acid, as in the Kjeldahl method for the determination of nitrogen, the clear solution was diluted and the copper deposited electrolytically. Check tests of the reagents proved them to be free from copper.

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NOTES.

Notes on Mr. Keen's Paper¹ on the Volumetric Determination of Zinc.

Mr. Keen disarms criticism by disclaiming any great originality for the method he describes. Unfortunately he has not selected the best of the old methods, and some of the things he advises are likely to cause trouble.

The methods given for preparing the ferrocyanide solution and for the titration are the very excellent ones described by Dr. Low.

The method given for standardizing is complicated and unreliable. A much simpler one is to partly dissolve a single large piece of high-grade spelter in dilute hydrochloric acid, dilute the solution so that it will contain about 5 grams of zinc per liter and determine the zinc by any reliable gravimetric method. I prefer to determine the zinc as pyrophosphate, as the method is simpler than most, and I have found it extremely accurate. If not more than three-quarters of the piece of spelter is dissolved the solution will contain nothing but zinc, and consequently needs no purification. Two or three liters can be made up at once and used as a standard for many months. If extreme accuracy is required, the zinc should be determined in weighed portions of the solution and weighed amounts be used for standardizing.

In the standardizing and actual analysis the volume of the solution, temperature, amount of free acid and of ammonium chloride, the indicator and the method of using it should be kept within very narrow limits or the results will be unreliable. The effect of ammonium chloride on the amount of ferrocyanide necessary is usually neglected but it is quite important.

Sampling Spelter.—The method proposed is very unreliable and likely to cause errors. It has been condemned by the International Committee at the Congress of Applied Chemistry at Rome. The method they recommend is by far the best, *i. e.*, to saw the slabs and use the sawdust for a sample. It is best to saw each slab entirely in two, it must at least be cut to the middle each time. An ordinary band saw, such as is used

¹ THIS JOURNAL, 30, 225.

for wood, answers perfectly; the saw should have rather fine teeth and be run at a high speed. The feed, of course, must be slow.

Analysis.—Common western spelter containing one or two per cent. of lead will not all dissolve in hydrochloric acid, and the residue is likely to contain both zinc and iron. It should be filtered out, dissolved in nitric acid, evaporated with sulphuric, the lead sulphate filtered out and the filtrate added to the main solution.

The methods of separation proposed are nearly all slow and the accuracy of some, at least, is very doubtful. The method¹ recommended by the Committee of this Society on Uniformity of Zinc Analysis is easier, quicker, simpler and far more accurate than the one proposed, and it is applicable to all zinciferous materials.

Aluminum Alloys.—In many cases the presence of aluminum does very seriously affect the ferrocyanide precipitation.

NEW JERSEY ZINC CO.,
71 BROADWAY, NEW YORK.

GEO. C. STONE.

The Detection and Identification of Manganese and Chromium in the Presence of Each Other.—“To the cold, dilute nitric or sulphuric acid solution of the substance or mixture to be tested is added one or two cubic centimeters of a silver nitrate solution of the ordinary concentration, then a relatively large amount (two to five grams) of solid potassium persulphate, and the whole carefully heated until the evolution of oxygen due to the decomposition of the persulphate is practically over. By this means the manganese is converted into permanganic acid and the chromium into chromic acid. The permanganate color shows itself first and is usually best seen during the first few moments of heating. In order to detect the chromium present (the chromate or dichromate color being usually obscured by the permanganate color), the cooled solution is shaken with one-fourth to one-third its volume of ether, hydrogen peroxide added in excess, and the mixture well shaken. This decomposes the permanganate with evolution of oxygen and converts the chromic acid into perchromic acid, which dissolves to a blue color in the excess of ether. Acetic ester may sometimes be used to advantage in place of ordinary ether.” The method is delicate, easily performed in an ordinary test-tube, and convenient in having no filtrations or fusions. In a course of Qualitative Analysis it may be tried either upon the original material or upon the proper group precipitate as one wishes. Halides should be absent and in case much manganese is present only small amounts of the substance analyzed should be taken, otherwise the manganese tends to be precipitated as manganese dioxide instead of being converted into permanganate.

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¹ THIS JOURNAL, 28, 262 (1907).